(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 319 624 A1**

(12) **EUROPEAN PATENT APPLICATION**

(21) Application number: 87310895.5

(51) Int. Cl.⁴: C07F 11/00 , C07F 15/06

(22) Date of filing: 10.12.87

(43) Date of publication of application: 14.06.89 Bulletin 89/24

(84) Designated Contracting States: DE FR GB IT NL

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(54) **Heterometallic thiocubanes and method of making them.**

(57) This invention relates to heterometallic thiocubane compositions containing the M^1_2 M^2_2 S_4 cluster core and methods of making them wherein M^1 is Re, V, Mo or W and M^2 is Co, Cr, Cu, Ni or Fe but preferably Co. More particularly, the invention relates to compositions of the formula M^2_2 M^2_2 $S_4 L^1_2$ L^2_2 L^3_2 wherein M^1 and M^2 are as above, L^1 is a bidentate sulfur or nitrogen bearing ligand (most preferably a dialkyldithiocarbamate), L^2 is optional but may be an O, N, P or S-containing monodentate donor ligand, and L^3 may be CO, a monodentate anion ligand such as a halide (preferably Cl), mercaptide or alkoxide, or another O, N, P, or S containing monodentate donor ligand. The compositions are suitable for making active hydrotreating catalysts.

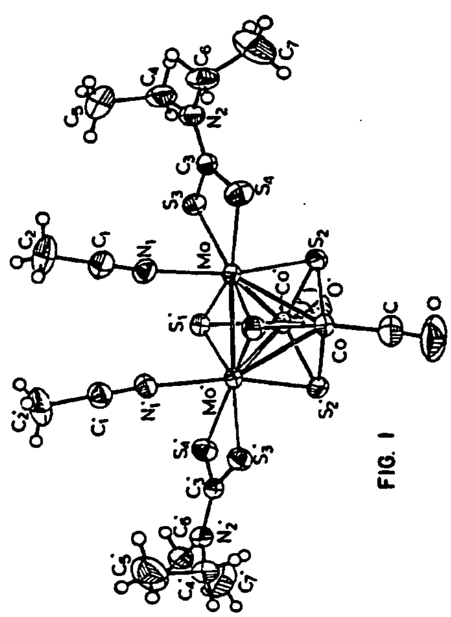

FIG. 1

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HETEROMETALLIC THIOCUBANES AND METHOD OF MAKING THEM

FIELD OF THE INVENTION

This invention relates to heterometallic thiocubane compositions containing the M^1_2 M^2_2 S_4 cluster core and methods of making them wherein M^1 is Re, V, Mo or W but preferably Mo or W; and M^2 is Co, Cr, Cu, Ni, or Fe but preferably Co. More particularly, the invention relates to compositions of the formula M^1_2 M^2_2 $S_4L^1_2$ L^2_2 L^3_2 wherein M^1 and M^2 are as above, L^1 is a bidentate sulfur and/or nitrogen bearing ligand (desirably a dithiolate and preferably a dialkyldithiocarbamate), L^2 is optional and may be an O, N, P, or S-containing monodentate donor ligand, and L^3 may be CO, a monodentate anion ligand such as a halide (preferably Cl), mercaptide or alkoxide, or another O, N, P, or S containing monodentate donor ligand. The compositions are suitable for making active hydrotreating catalysts.

BACKGROUND OF THE INVENTION

Thiocubane clusters containing a homometallic core M_4S_4 are known in the art. The thiocubane core is so-named because of its molecular architecture, i.e., two tetrahedra of metal and sulfur atoms interlock so that the metal atoms and bridging sulfurs occupy the alternate corners of a distorted or approximate cube. Homometallic thiocubane structures containing, e.g., Co, Fe, Mo, have been synthesized and discussed at length in the chemical literature. See, e.g., Mak et al, Angew. Chem. Int. Ed. Engl. 23 (1984), pp. 391-2; Shibahara et al, J. Am. Chem. Soc. (1984), 106, pp. 789-791; Chu et al, J. Am. Chem. Soc. (1982), v. 104, pp. 3409-3422 (and references cited therein) and Simon et al, J. Am. Chem. Soc. (1973), v. 95, pp. 2164-2174.

Compositions containing heterometallic thiocubane clusters have also been studied. The particular interest in the Fe_3MoS_4 cluster, because of its possible function as the biologically active part of nitrogenase, has led to the attempted synthesis of other similar compositions. See, e.g., Curtis et al, Inorg. Chem., v. 22, pp. 2661-2; Brunner et al, Agnew. Chem. Int. Ed. Engl 22 (1983), pg. 549; Brunner et al, J. Organometallic Chem., 240 (1982) C41-C44; Holm, Chem. Soc. Rev. (1981), v. 10, p. 455; and Armstrong et al., Inorg. Chem. (1982) v. 21, 1699-1701.

In addition to the interest shown in the bimetallic thiocubane cluster as a biologically active enzyme constituent, others have suggested that sulfided clusters containing molybdenum and a Group VIII metal, e.g., Fe, Co or Ni, may be useful as models in clarifying the somewhat poorly understood activity of hydrodesulfurization catalysts based on "sulfided" iron, cobalt, or nickel molybdates and tungstates on oxide supports. See, Curtis et al, supra, and the references cited therein; Gates et al, "Chemistry of Catalytic Processes", McGraw-Hill, New York (1979), pp. 390-445.

In the earlier syntheses of the thiocubane core, the approach was typically "spontaneous assembly". See, Holm, supra. Later work in homometallic transition metal sulfide chemistry led to smaller clusters which could be considered fragments of the thiocubane unit. These fragments, e.g., $Cp_2M_2S_4$ and related compounds (where Cp represents the cylcopentadienyl ligand), are potential building blocks for heteronuclear thiocubane clusters and can be used to form clusters with $M_2M'_2$ $(\mu^3\text{-}S)_4$ cores. See, the two Brunner et al articles, supra.

None of the prior art shows the synthesis of M^1_2 M^2_2 $(\mu^3\text{-}S)_4L^1_2$ L^2_2 L^3_2 where M^1 is Re, V, Mo or W, M^2 is Co, Cr, Cu, Ni or Fe, L^1 is a bidentate sulfur and/or nitrogen bearing ligand, and L^2 is optional but may be an S, N, P, or O monodentate donor ligand, e.g., a solvent or other Lewis base molecule, and L^3 may be CO, a monodentate anion ligand such as a halide (preferably Cl), mercaptide or alkoxide, or another O, N, P, or S containing monodentate donor ligand.

SUMMARY OF THE INVENTION

This invention deals with compositions containing heterometallic thiocubane nuclei. In particular the compositions have the generalized formula M^1_2 M^2_2 $(\mu^3\text{-}S)_4L^1_2$ L^2_2 L^3_2 where M^1 is Re, V, Mo or W (but preferably Mo or W); M^2 is Co, Cr, Cu, Ni or Fe (but preferably Co); L^1 is a bidentate sulfur and/or nitrogen bearing ligand such as dithiolates and particularly xanthate, o-aminobenzenethiolate, dithiophosphinate, dithiophosphate (but preferably a dithiocarbamate (S_2CNR_2) where R is independently H, or a hydrocarbyl such as methyl, ethyl, propyl, butyl, phenyl or mixtures of such groups, but preferably ethyl); L^2 is optional but may be an S, N, P, or O monodentate donor ligand, e.g., a solvent or other Lewis base molecule such as a pyridine, ether or phosphine ligand (but is preferably acetonitrile); and L^3 may be CO, a monodentate anion ligand such as a halide (preferably Cl), mercaptide or alkoxide, or another O, N, P, or S containing monodentate donor ligand.

The heterometallic thiocubane composition is preferably synthesized using novel dimeric neutral complexes of, e.g., tungsten, sulfide and dialkyldithiocarbamates.

The heterometallic thiocubane compositions have various uses but are especially useful in the preparation of catalysts for hydrotreating hydrocarbons containing sulfur-bearing compounds.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a molecular depiction of one composition, i.e., $Mo_2Co_2(\mu^3\text{-}S)_4((C_2H_5)_2NCS_2)_2\text{-}(CO)_2(CH_3CN)_2$, within the scope of the invention.

Figure 2 is a molecular depiction of one composition, i.e., $Mo_2Cu_2(\mu^3\text{-}S)_4((C_4C_9)_2NCS_2)_2\text{-}(Cl)_2$, within the scope of the invention.

DESCRIPTION OF THE INVENTION

The invention, as noted above, is generically a composition of matter containing certain heterometallic thiocubane clusters. The broad formula for the composition is:

$$M^1_2\, M^2_2\, (\mu^3\text{-}S)_4 L^1_2\, L^2_2\, L^3_2$$

where:

M^1 is Re, V, Mo or W; preferably Mo or W;

M^2 is Co, Cr, Cu, Ni or Fe; preferably Co;

L^1 is a bidentate sulfur and/or nitrogen-bearing ligand such as amino benzene thiolate or dithiolates, particularly xanthate, dithiophosphinate, dithiophosphate, dithiocarbamate, etc.; preferably dithiocarbamate (S_2CNR_2) where R is independently an H or a hydroarbyl, such as methyl, ethyl, propyl, butyl, phenyl, most preferably ethyl; other suitable ligands include those having the formula: $C_6H_4SNH_2$, SCH_2CH_2S, and $CH_3NHCH_2(CH_3)_2S$.

L^2 is optional but may be a monodentate S, N, P, or O donor ligand, e.g., a solvent or other Lewis base molecule such as a pyridine, ether or phosphine but preferalβly acetonitrile. The thiocubane core is, however, stable without the presence of L^2.

L^3 may be CO, a monodentate anion ligand such as a halide (preferably Cl), mercaptide or alkoxide, or another O, N, P, or S containing monodentate donor ligand.

The preferred method of synthesizing the inventive heterometallic thiocubanes involves the addition of a generally stoichiometric amount of a low valent complex based upon one of the "M^2" metals Co, Cr, Cu, Ni or Fe such as $Co_2(CO)_8$, $Ni(CO)_4$ SO_2CuCl to a solution or slurry of $M^1_2\, S_4 L^1_2$. The metal M^1, as above, may be Re, V, Mo or W although preferably Mo or W. To achieve the goal of including substantial sulfur in a hydrodesulfurization catalyst using the inventive compound as a precursor, the bidentate ligand should be a mono- or a dithiolate. Although a large number of such ligands may be used, e.g., amino benzene thiolates, xanthates, dithiophosphinates, dithiophosphates, the preferred ligand is a dithiocarbamate of the formula S_2CNR_2 wherein R is independently H or a hydrocarbyl or alkyl of C_1 to C_{12}. The diethyl form is especially preferred.

These two materials may be placed together in a suitable solvent which may, by default, act as the monodentate ligand L^2 (or L^3) above. Acetonitrile is especially useful.

These "inorganic synthons" obviously act as building blocks to the final inventive heterometallic thiocubane composition. The reaction usually proceeds at room temperature with no heat input required.

Although the syntheses of a majority of the materials used in producing the thiocubanes are known, the production of one such fragment is not, i.e., $W_2S_4(S_2CNR_2)_2$ where R is an H or a C_1 to C_{12} hydrocarbyl group.

These fragments may be made by gently heating $(NEt_4)_2W_2S_{12}$ in an acetonitrile solution, or other aprotic solvent, in the presence of $R_2NCS_2{}_2^-$, NH_4^+, and $P(C_6H_5)_3$. When making the tungsten-based compound, the product is found in and may be isolated from an orange slurry.

This process may be used to produce compounds of the formula:

$$W_2S_4(S_2CNR^1R^2)_2$$

where R^n = H, an alkyl of 1 to 12 carbon atoms, and aryl groups of 6 to 12 carbamates. R^1 need not be the same as R^2.

and also

$$W_2S_4(S_2COR)_2$$

where R are alkyl or aryl groups of the type discussed above.

Having thus described the invention in detail, following are a number of examples which further delineate the invention. These examples are not intended to be limiting in any manner to the invention claimed below.

EXAMPLE 1

Production of $(NEt_4)_2W_2S_{12}$

Excess sulfur (1.18 g, 4.60 mmol) was added to a stirred solution of $(NH_4)_2WS_4$ (3.20 g, 9.20 mmol) in 50 mL DMF and heated two hours at 110° while purging slowly with Ar. The resulting orange-red solution was allowed to cool, excess NEt_4Br (2.55 g, 12.1 mmol) was added, and the mixture heated at 110° for an additional 60 min. Volatiles were removed at 60° under vacuum, leaving an oily orange-red solid. Repeated recrystallization of the product from acetonitrile with intermittent washings with toluene, methanol, and diethylether yielded 3.85 g (83%) of red crystalline $(NEt_4)_2W_2S_{12}$.

Anal. Calcd for $C_{16}H_{40}N_2S_{12}W_2$: C, 18.97, H, 3.98, N, 2.77, S, 37.98, W, 36.30. Found: C, 19.00, H, 3.86, N, 2.68, S, 37.44, W. 36.25. IR: 3000 (w), 2980 (w), 1474 (s), 1445 (m), 1432 (m), 1417 (w), 1390 (m), 1385 (m), 1312 (m), 1184 (w), 1168 (s), 1065 (w), 1049 (w), 996 (s), 777 (s), 506 (vs), 495 (sh), 460 (w), 423 (m), 414 (m), 405 (sh), 380 (m), 371 (w), 294 (m) cm^{-1}. FAB-MS (in tetramethylenesulfone) m/e for parent negative ion $[(NEt_4)W_2S_{12}]^-$(rel. abundance calc, exp): 878 (19,25): 879 (23,28); 880 (62,62); 881 (45,51); 882 (100,100), 883 (55,61); 884 (96,98); 885 (32,42); 886 (60,62); 887 (14,19); 888 (21,25). ^{183}W NMR (1.11g $(NEt_4)_2W_2S_{12}$ in 2.2 mL DMF and 0.7 mL DMF-d_7): 2131.

EXAMPLE 2

Production of $W_2S_4(S_2CNEt_2)_2$

An acetonitrile solution of (50 ml) containing PPh_3 (0.8 g, 3 mmol), NH_4PF_6 (0.48 g, 2.9 mmol), $Na(S_2CNEt_2)\cdot 3H_2O$ (0.35 g, 1.6 mmol), and $(NEt_4)_2W_2S_{12}$ (0.508 g, 0.502 mmol) made according to Example 1 was heated at 75° for 45 min., resulting in the formation of an orange slurry. Volatiles were removed in vacuo, leaving a mixture of orange and white solids. The mixture was cooled to 0° and washed with 2 x 15 ml CH_3OH, 2 x 20 ml acetone, and 3 x 20 ml ether. The resulting orange powder was dried under vacuum. Yield was 300 mg (75%). Air-stable $W_2S_4(S_2CNEt_2)_2$ is very slightly soluble in DMF and hot acetonitrile.

Anal. Calcd. for $C_{10}H_{20}N_2S_8W_2$: C, 15.16; H, 2.54; N, 3.53; S, 32.36. Found: C, 14.94; H, 2.30; N, 3.57; S, 32.34. IR: 2980 (w), 2935 (w), 1530 (s), 1456 (m), 1440 (m), 1382 (w), 1358 (m), 1297 (w), 1281 (s), 1201 (m), 1152 (m), 1098 (w), 1077 (m), 1006 (w), 996 (w), 908 (w), 847 (w), 779 (w), 527 (s), 519 (s), 445 (m), 371 (m), 323 (m) cm^{-1}. Field desorption mass spectrum m/e for parent $W_2S_8C_{10}H_{20}N_2$, relative abundance (calc., exp.): 788 (21,21); 789 (26,24); 790 (66,68); 791 (46,43); 792 (100,100); 793 (52,60); 794 (89,79); 795 (26,32); 796 (50,41).

EXAMPLE 3

Production of $W_2S_4(S_2CN(i\text{-}C_4H_9)_2)_2$

The procedure of Example 2 was repeated using $Na(S_2CN(i\text{-}C_4H_9)_2)$ instead of the diethyl analog. The resulting product had characteristic IR bands at 535, 523, 448, 372 and 328 cm^{-1} (all ± 5 cm^{-1}).

EXAMPLE 4

Production of $(Et_2NCS_2)_2(MeCN)_2W_2(\mu^3\text{-}S)_4Co_2(CO)_2$

In an inert atmosphere glove box, solid $Co_2(CO)_8$ (0.505 g, 1.48 mmol) was added over a period of five minutes to a stirred orange slurry of $W_2S_4(S_2CNEt_2)_2$ (1.175 g, 1.49 mmol) in 80 ml acetonitrile as made according to Example 2. The solution darkened to a brown-black slurry as gas was evolved. The mixture was stirred 2 hours and then filtered. The filtrate was concentrated to ca. 60 ml and placed in a -10° freezer overnight. Black crystalline $(Et_2NCS_2)_2(MeCN)_2W_2(\mu^3\text{-}S)_4Co_2(CO)_2$ was filtered from the dark solution on a sintered-glass Schlenk filter, washed with 8 mL acetonitrile, and dried in vacuo. Yield was 650 mg (42%).

Anal. Calcd. for $C_{16}H_{26}N_4O_2S_8Co_2W_2$: C, 18.33; H, 2.50; N, 5.34; W, 35.07. Found: C, 18.03; H, 2.49; N, 5.27; W, 35.27. IR (KBr pellet): 1961(s), 1938(s), 1505(s), 1456(w), 14.36(m), 1358(w), 1300(w), 1273(m), 1209(m), 1147(m), 1095(w), 1075(m), 915(w), 847(w), 783(w), 521(m), 394(w), and 368(w) cm^{-1}.

EXAMPLE 5

Production of $(Et_2NCS_2)_2(MeCN)_2Mo_2(\mu^3\text{-}S)_4CO_2(CO)_2$

In an inert atmosphere glove box, solid $Co_2(CO)_8$ (sublimed, 0.111 g, 0.324 mmoles) was added over a period of several minutes to a stirred slurry of red-brown $Mo_2S_4(S_2CNEt_2)_2$ (0.200 g, 0.324 mmoles) in 20 ml dry CH_3CN (distilled from CaH_2). This material was made via the process disclosed in Miller et al, J. Am. Chem. Soc. (1980), pp. 5104-5106. The solution darkened rapidly with gentle evolution of carbon monoxide. After stirring for two hours, the solution was filtered and the dark black-brown filtrate was concentrated under vacuum to a volume of 5 ml. The mixture was then allowed to stand 18 hours. Black crystalline $(Et_2NCS_2)_2(MeCN)_2Mo_2(\mu^3\text{-}S)_4Co_2(CO)_2$ was filtered on a medium porosity sintered glass frit, and dried in vacuo. Yield was 200 mg (71%).

IR spectrum (KBr pellet): 1983(s), 1960(s), 1505(s), 1465(m), 1442(m), 1385(w), 1368(m), 1310(w), 1280(s), 1220(m), 1152(m), 1103(w), 1083(m), 1008(w), 976(w), 922(w), 851(w), 790(w), 578(w), 521(m), 498(w), 436(vw), 401(w), 371(m). Anal. Calcd. for $C_{16}H_{26}N_4O_2S_8Co_2Mo_2$: C, 22.02; H, 3.00; N, 6.42; Mo, 21.99; Co, 13.51. Found C, 18.59; H, 2.83; N, 5.11; Mo, 21.10; Co, 14.22.

A single crystal x-ray diffraction study was carried out on the product. The structure is illustrated in Figure 1 and the x-ray structure factors are given in Table 1. The molecule contains a $Co_2Mo_2(\mu_3\text{-}S)_4$ core. The four metal atoms are joined by six metal-metal bonds forming an approximate tetrahedron of C_{2v} symmetry. Each triangular face of the tetrahedron is capped by a sulfur, to form the overall "thiocubane" core. Each cobalt is further bonded to a single terminal CO. The coordination environment about the cobalt atoms (discounting the M-M bonds) very nearly tetrahedral. Each molybdenum atom is bound to two dithiocarbamate sulfur atoms and to the nitrogen of an acetonitrile molecule, in addition to three capping "μ^3-S" atoms. The coordination environment about the molybdenum atoms is distorted octahedral. The molecule resides on a crystallographic C_2 axis which bisects the Mo-Mo′ and Co-Co′ bonds.

TABLE 1

$MO_2CO_2S_4(CO)_2(NCCH_3)_2[S_2CH(C_2H_5)_2]_2$

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
2	0	0	4951	4967	20	4	0	202	192
4	0	0	1638	1654	24	4	0	295	289
6	0	0	2661	2633	1	5	0	2267	2247
8	0	0	5917	5956	3	5	0	2207	2178
10	0	0	4832	4831	5	5	0	206	190
12	0	0	2120	2099	7	5	0	1914	1887
14	0	0	641	656	9	5	0	1668	1658
16	0	0	679	675	11	5	0	1846	1841
18	0	0	1518	1511	13	5	0	674	685
20	0	0	1127	1119	15	5	0	212	102
22	0	0	637	626	17	5	0	776	776
24	0	0	566	543	19	5	0	728	719
1	1	0	1089	1082	21	5	0	623	623
3	1	0	968	959	23	5	0	217	231
5	1	0	793	797	0	6	0	3088	3042
7	1	0	604	618	2	6	0	2843	2796
9	1	0	1777	1775	4	6	0	362	366
11	1	0	476	462	6	6	0	668	675
13	1	0	187	206	8	6	0	1851	1839
15	1	0	451	439	10	6	0	2127	2112
17	1	0	274	263	12	6	0	1091	1107
19	1	0	520	526	14	6	0	489	491
27	1	0	244	222	18	6	0	692	707
0	2	0	1656	1650	20	6	0	564	558
2	2	0	2666	2658	22	6	0	574	579
4	2	0	836	829	24	6	0	502	504
6	2	0	344	349	1	7	0	150	102
8	2	0	1955	1974	3	7	0	802	807
10	2	0	1651	1632	5	7	0	599	596
12	2	0	827	802	7	7	0	320	314
14	2	0	289	294	9	7	0	316	307
16	2	0	115	126	11	7	0	643	668
18	2	0	590	594	13	7	0	1342	1364
20	2	0	503	490	15	7	0	258	264
22	2	0	353	349	17	7	0	527	523
24	2	0	268	278	21	7	0	486	484
1	3	0	3046	3044	23	7	0	268	269
3	3	0	974	932	0	8	0	192	184
5	3	0	828	811	2	8	0	333	309
7	3	0	1481	1509	4	8	0	510	526
9	3	0	1836	1815	6	8	0	609	595
11	3	0	353	338	8	8	0	292	272
13	3	0	730	726	10	8	0	226	188
15	3	0	479	461	12	8	0	746	752
17	3	0	935	926	14	8	0	132	145
19	3	0	708	713	16	8	0	327	335
23	3	0	129	139	18	8	0	283	277
0	4	0	4682	4724	20	8	0	563	562
2	4	0	1863	1869	22	8	0	476	470
4	4	0	662	642	1	9	0	1706	1695

TABLE 1-continued

Mo2Co2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
6	4	0	359	349	3	9	0	998	989
8	4	0	2656	2596	5	9	0	404	409
10	4	0	2096	2117	7	9	0	1276	1284
12	4	0	346	339	9	9	0	1452	1431
14	4	0	259	263	11	9	0	884	873
18	4	0	622	612	13	9	0	164	185
15	9	0	562	566	−10	2	1	1180	1147
17	9	0	655	670	−8	2	1	811	801
19	9	0	490	496	−6	2	1	1215	1254
0	10	0	820	824	−4	2	1	1016	972
2	10	0	451	473	−2	2	1	2168	2199
8	10	0	260	267	0	2	1	477	460
10	10	0	291	312	2	2	1	2305	2244
14	10	0	248	267	4	2	1	659	649
16	10	0	272	298	6	2	1	860	891
1	11	0	1141	1134	8	2	1	981	1021
3	11	0	1036	1046	10	2	1	174	188
5	11	0	182	179	12	2	1	387	395
7	11	0	629	642	16	2	1	675	654
9	11	0	604	610	18	2	1	1529	1476
11	11	0	1066	1064	20	2	1	586	574
13	11	0	1007	1006	22	2	1	517	493
15	11	0	177	200	26	2	1	807	757
0	12	0	399	406	−23	3	1	210	219
2	12	0	372	397	−19	3	1	383	356
4	12	0	214	239	−17	3	1	395	391
6	12	0	372	360	−13	3	1	241	224
8	12	0	429	453	−11	3	1	623	614
10	12	0	314	295	−9	3	1	1252	1209
12	12	0	349	360	−7	3	1	111	104
1	13	0	210	216	−5	3	1	740	733
3	13	0	317	317	−3	3	1	618	611
5	13	0	249	260	−1	3	1	2024	1997
9	13	0	193	207	1	3	1	221	231
−25	1	1	348	338	3	3	1	267	254
−23	1	1	389	394	5	3	1	440	431
−21	1	1	666	676	7	3	1	443	433
−19	1	1	1170	1178	11	3	1	306	317
−17	1	1	696	695	13	3	1	210	220
−15	1	1	515	525	15	3	1	448	444
−13	1	1	1186	1211	17	3	1	232	246
−11	1	1	3331	3248	19	3	1	305	288
−9	1	1	3748	3860	21	3	1	442	428
−7	1	1	706	706	25	3	1	160	178
−5	1	1	465	472	−22	4	1	266	275
−3	1	1	3777	3637	−20	4	1	569	598
−1	1	1	5915	5903	−18	4	1	675	677
1	1	1	2840	2847	−14	4	1	434	436
3	1	1	1779	1770	−12	4	1	1238	1250
7	1	1	2125	2096	−10	4	1	1678	1694

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
9	1	1	2700	2710	−8	4	1	1310	1254
11	1	1	1511	1547	−6	4	1	204	236
13	1	1	939	974	−4	4	1	2233	2150
15	1	1	487	497	−2	4	1	3028	3001
17	1	1	963	953	0	4	1	2891	2844
19	1	1	958	934	2	4	1	612	591
21	1	1	574	576	4	4	1	1443	1429
23	1	1	616	585	6	4	1	1894	1888
−22	2	1	467	475	8	4	1	2844	2821
−18	2	1	241	272	10	4	1	1548	1543
−14	2	1	487	519	14	4	1	368	360
−12	2	1	674	663	16	4	1	1436	1410
18	4	1	1198	1175	−7	7	1	659	674
20	4	1	873	860	−5	7	1	583	576
22	4	1	358	361	−3	7	1	1042	1052
24	4	1	284	250	−1	7	1	1219	1194
−25	5	1	349	351	1	7	1	1211	1194
−23	5	1	262	252	3	7	1	659	661
−21	5	1	392	383	5	7	1	530	517
−19	5	1	933	954	7	7	1	804	780
−17	5	1	458	471	9	7	1	1433	1392
−13	5	1	1029	1032	11	7	1	1079	1061
−11	5	1	2675	2685	13	7	1	523	529
−9	5	1	3071	3038	15	7	1	241	251
−7	5	1	698	718	17	7	1	618	599
−5	5	1	440	460	19	7	1	825	793
−3	5	1	2203	2228	21	7	1	869	836
−1	5	1	3894	3899	23	7	1	201	211
1	5	1	2799	2787	−22	8	1	404	441
3	5	1	443	452	−18	8	1	331	349
5	5	1	533	513	−16	8	1	476	500
7	5	1	1367	1332	−14	8	1	479	531
9	5	1	1937	1932	−10	8	1	617	610
11	5	1	920	911	−8	8	1	346	336
13	5	1	1336	1312	−6	8	1	186	184
15	5	1	658	660	−4	8	1	368	373
17	5	1	706	688	−2	8	1	457	444
19	5	1	609	588	0	8	1	524	514
21	5	1	514	506	2	8	1	552	553
23	5	1	571	541	4	8	1	936	943
−24	6	1	406	423	6	8	1	468	486
−22	6	1	710	741	8	8	1	901	884
−20	6	1	504	528	12	8	1	325	305
−18	6	1	177	177	14	8	1	285	271
−14	6	1	992	1032	16	8	1	743	718
−12	6	1	1412	1521	18	8	1	597	508
−10	6	1	1189	1170	20	8	1	184	162
−8	6	1	1064	1108	−15	9	1	203	205
−6	6	1	211	232	−13	9	1	513	541
−4	6	1	1519	1517	−7	9	1	346	331

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−2	6	1	1516	1516	−5	9	1	593	593
0	6	1	1527	1482	−3	9	1	834	827
2	6	1	2328	2331	3	9	1	191	172
6	6	1	981	956	5	9	1	1430	1420
8	6	1	625	617	7	9	1	939	917
10	6	1	1291	1252	9	9	1	417	379
12	6	1	1108	1082	11	9	1	591	591
14	6	1	278	281	13	9	1	614	603
16	6	1	212	193	15	9	1	1013	1017
18	6	1	185	195	17	9	1	658	645
20	6	1	308	328	19	9	1	629	613
−23	7	1	430	418	−18	10	1	303	315
−21	7	1	386	403	−14	10	1	718	735
−19	7	1	228	254	−12	10	1	1051	1037
−15	7	1	434	468	−10	10	1	1014	1007
−13	7	1	668	701	−8	10	1	976	907
−11	7	1	663	727	−6	10	1	187	167
−9	7	1	550	562	−4	10	1	1017	1044
−2	10	1	1554	1571	8	0	2	3478	3537
0	10	1	1399	1407	10	0	2	1197	1186
2	10	1	1325	1328	12	0	2	858	872
6	10	1	1119	1107	14	0	2	617	614
8	10	1	999	1005	16	0	2	1730	1681
10	10	1	1316	1312	18	0	2	1023	1011
12	10	1	723	740	20	0	2	374	366
14	10	1	276	258	22	0	2	271	297
16	10	1	449	441	24	0	2	446	448
18	10	1	548	549	26	0	2	725	724
−11	11	1	251	281	−25	1	2	267	280
−9	11	1	414	441	−19	1	2	312	296
−7	11	1	307	299	−17	1	2	675	672
−1	11	1	352	378	−15	1	2	607	590
1	11	1	517	503	−11	1	2	191	133
3	11	1	456	466	−9	1	2	1751	1795
5	11	1	203	194	−7	1	2	987	1033
9	11	1	200	236	−5	1	2	553	555
11	11	1	497	510	−3	1	2	1603	1623
13	11	1	509	499	−1	1	2	398	390
15	11	1	187	208	1	1	2	814	777
−12	12	1	774	752	3	1	2	2183	2185
−10	12	1	381	373	5	1	2	434	429
−8	12	1	236	236	7	1	2	1374	1382
−4	12	1	419	425	9	1	2	895	900
−2	12	1	485	499	11	1	2	537	562
0	12	1	622	630	13	1	2	285	292
2	12	1	885	890	15	1	2	647	649
4	12	1	437	445	17	1	2	1746	1711
8	12	1	251	274	19	1	2	685	685
10	12	1	772	763	21	1	2	638	625
12	12	1	721	725	25	1	2	748	709

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2									
H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−9	13	1	263	288	−26	2	2	279	301
−5	13	1	279	313	−24	2	2	373	380
−3	13	1	680	707	−22	2	2	311	306
−1	13	1	425	441	−20	2	2	282	280
1	13	1	436	428	−16	2	2	365	364
5	13	1	704	711	−14	2	2	896	881
7	13	1	782	782	−12	2	2	753	762
9	13	1	556	571	−10	2	2	760	713
−26	0	2	434	421	−8	2	2	152	108
−24	0	2	817	822	−4	2	2	1313	1356
−22	0	2	1096	1107	−2	2	2	1413	1419
−20	0	2	1591	1569	0	2	2	1573	1560
−18	0	2	592	595	2	2	2	1180	1176
−16	0	2	1418	1397	4	2	2	494	467
−14	0	2	1816	1784	6	2	2	450	466
−12	0	2	4108	4013	8	2	2	823	831
−10	0	2	2924	2818	10	2	2	863	864
−8	0	2	353	381	12	2	2	866	871
−6	0	2	2451	2405	14	2	2	145	125
−4	0	2	3143	3043	16	2	2	494	484
−2	0	2	4371	4488	18	2	2	778	759
0	0	2	2190	2190	20	2	2	516	503
2	0	2	2088	2068	22	2	2	163	175
6	0	2	1789	1769	24	2	2	228	211
26	2	2	367	333	−1	5	2	2346	2353
−25	3	2	277	297	1	5	2	655	661
−17	3	2	1061	1079	3	5	2	653	648
−15	3	2	860	876	5	5	2	1455	1442
−13	3	2	435	426	7	5	2	1493	1475
−11	3	2	299	276	9	5	2	1106	1105
−9	3	2	1479	1375	11	5	2	561	560
−7	3	2	2320	2464	13	5	2	604	595
−5	3	2	1495	1466	15	5	2	1004	900
−3	3	2	558	570	17	5	2	452	437
−1	3	2	137	148	19	5	2	693	602
1	3	2	1268	1225	23	5	2	331	333
3	3	2	288	314	−24	6	2	467	463
5	3	2	900	903	−22	6	2	439	430
7	3	2	2724	2737	−20	6	2	478	489
9	3	2	1623	1622	−16	6	2	580	609
11	3	2	441	464	−14	6	2	760	819
13	3	2	297	284	−12	6	2	831	896
15	3	2	1551	1502	−10	6	2	1085	1119
17	3	2	2237	2182	−8	6	2	154	108
19	3	2	1362	1324	−6	6	2	222	189
21	3	2	656	641	−4	6	2	509	511
25	3	2	1024	980	−2	6	2	946	953
−24	4	2	226	235	0	6	2	1542	1527
−22	4	2	378	371	2	6	2	706	703
−20	4	2	850	876	4	6	2	1466	1466

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−18	4	2	605	623	6	6	2	541	523
−16	4	2	243	212	8	6	2	1252	1221
−14	4	2	445	419	10	6	2	1233	1211
−12	4	2	1578	1551	12	6	2	1506	1480
−10	4	2	1672	1643	14	6	2	251	280
−8	4	2	423	418	16	6	2	606	573
−6	4	2	753	751	18	6	2	1043	1028
−2	4	2	2151	2127	20	6	2	786	759
0	4	2	564	560	22	6	2	323	316
2	4	2	858	826	−23	7	2	370	381
4	4	2	1584	1554	−21	7	2	328	331
6	4	2	774	766	−17	7	2	346	353
8	4	2	529	523	−13	7	2	352	360
10	4	2	261	265	−11	7	2	435	447
12	4	2	865	863	−9	7	2	1014	1042
14	4	2	1066	1029	−7	7	2	1403	1444
16	4	2	241	239	−3	7	2	216	234
20	4	2	179	168	−1	7	2	273	280
22	4	2	322	298	1	7	2	1849	1856
−25	5	2	338	347	3	7	2	448	447
−23	5	2	573	571	5	7	2	225	196
−21	5	2	514	526	7	7	2	661	658
−19	5	2	297	301	11	7	2	238	229
−15	5	2	763	780	13	7	2	156	134
−13	5	2	1611	1586	15	7	2	674	664
−11	5	2	1126	1121	17	7	2	781	758
−9	5	2	956	936	19	7	2	440	398
−7	5	2	216	207	21	7	2	173	132
−5	5	2	1635	1694	−22	8	2	171	168
−3	5	2	1705	1749	−20	8	2	268	255
−18	8	2	369	385	−11	11	2	620	618
−16	8	2	408	423	−9	11	2	510	494
−14	8	2	477	519	−7	11	2	297	304
−12	8	2	263	239	−5	11	2	514	535
−10	8	2	486	467	−3	11	2	676	699
−8	8	2	249	213	−1	11	2	715	732
−6	8	2	954	925	1	11	2	1112	1124
−4	8	2	945	930	3	11	2	258	276
−2	8	2	379	374	5	11	2	411	416
0	8	2	490	493	7	11	2	356	354
4	8	2	1350	1324	9	11	2	762	783
6	8	2	1093	1073	11	11	2	538	554
8	8	2	1065	1056	13	11	2	151	73
10	8	2	1017	1025	−4	12	2	221	262
12	8	2	179	148	0	12	2	212	243
14	8	2	812	811	2	12	2	191	212
16	8	2	783	784	4	12	2	274	270
18	8	2	831	826	6	12	2	178	155
20	8	2	741	710	8	12	2	330	340
−21	9	2	272	277	10	12	2	577	582

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−17	9	2	219	211	12	12	2	180	170
−15	9	2	552	598	−9	13	2	248	226
−13	9	2	671	683	−5	13	2	283	272
−11	9	2	681	650	−3	13	2	439	460
−9	9	2	227	232	−1	13	2	422	408
−7	9	2	509	506	1	13	2	199	205
−5	9	2	1138	1150	5	13	2	183	233
−3	9	2	1134	1146	7	13	2	218	243
−1	9	2	827	832	−27	1	3	436	451
1	9	2	255	242	−25	1	3	783	770
3	9	2	724	702	−23	1	3	654	644
5	9	2	913	926	−21	1	3	626	622
7	9	2	1121	1109	−17	1	3	1004	1061
9	9	2	782	771	−15	1	3	951	961
11	9	2	420	409	−13	1	3	943	904
13	9	2	395	373	−11	1	3	723	699
15	9	2	733	716	−9	1	3	353	341
17	9	2	820	793	−7	1	3	237	287
19	9	2	582	578	−5	1	3	747	682
−14	10	2	254	248	−3	1	3	671	710
−10	10	2	264	239	−1	1	3	1082	1117
−8	10	2	194	223	1	1	3	543	529
−6	10	2	584	618	3	1	3	1887	1846
−4	10	2	1085	1096	5	1	3	134	143
−2	10	2	371	334	7	1	3	1258	1261
0	10	2	176	151	9	1	3	373	367
2	10	2	751	756	11	1	3	409	404
4	10	2	1594	1595	15	1	3	1038	1049
6	10	2	1032	1032	17	1	3	439	441
8	10	2	230	240	23	1	3	393	391
10	10	2	252	251	25	1	3	444	426
12	10	2	864	850	−26	2	3	724	696
14	10	2	900	876	−24	2	3	149	187
16	10	2	333	348	−22	2	3	370	387
−15	11	2	546	509	−20	2	3	829	822
−13	11	2	799	778	−18	2	3	1804	1859
−16	2	3	1418	1403	10	4	3	567	570
−14	2	3	195	202	12	4	3	782	779
−12	2	3	1050	1008	14	4	3	1446	1425
−10	2	3	2267	2126	16	4	3	1572	1556
−8	2	3	2458	2356	18	4	3	931	931
−6	2	3	595	631	22	4	3	219	202
−2	2	3	324	323	24	4	3	654	616
0	2	3	803	797	−25	5	3	610	616
2	2	3	101	107	−23	5	3	422	428
4	2	3	140	162	−21	5	3	558	502
6	2	3	2646	2666	−19	5	3	197	155
8	2	3	2329	2354	−17	5	3	777	829
10	2	3	834	853	−15	5	3	457	481
12	2	3	704	709	−13	5	3	171	160

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
14	2	3	1288	1274	−11	5	3	486	492
16	2	3	2250	2213	−9	5	3	165	178
18	2	3	1159	1125	−7	5	3	419	447
20	2	3	889	880	−5	5	3	712	723
22	2	3	143	132	−3	5	3	283	275
24	2	3	777	741	−1	5	3	165	131
−21	3	3	370	351	1	5	3	606	608
−19	3	3	524	541	3	5	3	1573	1584
−17	3	3	193	148	5	5	3	398	410
−15	3	3	291	285	7	5	3	438	431
−13	3	3	341	330	9	5	3	169	169
−11	3	3	395	390	11	5	3	367	358
−9	3	3	166	140	13	5	3	411	388
−7	3	3	660	643	15	5	3	367	330
−5	3	3	754	755	17	5	3	323	284
−3	3	3	982	939	19	5	3	257	244
3	3	3	941	941	23	5	3	238	219
5	3	3	1319	1307	−24	6	3	386	394
7	3	3	319	343	−22	6	3	594	608
9	3	3	944	935	−20	6	3	251	252
13	3	3	663	654	−18	6	3	343	352
17	3	3	779	769	−16	6	3	176	217
19	3	3	593	591	−14	6	3	906	970
25	3	3	224	229	−12	6	3	674	734
−26	4	3	664	657	−10	6	3	1084	1113
−24	4	3	560	555	−8	6	3	503	541
−22	4	3	249	237	−6	6	3	854	870
−20	4	3	320	352	−4	6	3	1210	1212
−18	4	3	1169	1216	−2	6	3	601	610
−16	4	3	1515	1588	0	6	3	334	335
−14	4	3	1252	1273	2	6	3	700	680
−12	4	3	459	467	4	6	3	857	849
−10	4	3	1066	1048	8	6	3	484	486
−8	4	3	2465	2499	10	6	3	312	209
−6	4	3	2067	2172	12	6	3	956	939
−4	4	3	1435	1484	16	6	3	520	522
−2	4	3	1332	1311	18	6	3	176	139
0	4	3	1023	1009	20	6	3	316	275
2	4	3	928	949	22	6	3	355	354
4	4	3	2318	2358	−23	7	3	245	274
6	4	3	2262	2294	−21	7	3	173	186
8	4	3	1473	1477	−19	7	3	546	575
−17	7	3	655	706	19	9	3	462	452
−15	7	3	628	655	−18	10	3	280	306
−13	7	3	334	354	−16	10	3	568	603
−11	7	3	385	405	−14	10	3	910	906
−9	7	3	672	650	−12	10	3	517	506
−7	7	3	966	976	−8	10	3	402	406
−5	7	3	191	161	−6	10	3	907	909
−3	7	3	364	373	−4	10	3	751	762

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2									
H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−1	7	3	201	185	−2	10	3	477	495
1	7	3	176	195	0	10	3	307	301
3	7	3	243	194	2	10	3	441	442
5	7	3	670	668	4	10	3	757	775
7	7	3	753	751	6	10	3	528	511
9	7	3	1097	1103	8	10	3	503	517
11	7	3	448	432	12	10	3	392	393
13	7	3	428	398	14	10	3	517	514
15	7	3	705	687	16	10	3	358	367
17	7	3	961	938	−13	11	3	329	304
19	7	3	670	648	−11	11	3	323	280
−20	8	3	278	297	−7	11	3	369	344
−18	8	3	734	780	−5	11	3	675	671
−16	8	3	683	726	−3	11	3	427	436
−12	8	3	307	291	1	11	3	395	401
−10	8	3	733	713	3	11	3	733	741
−8	8	3	1142	1086	5	11	3	549	551
−6	8	3	493	476	9	11	3	313	327
−4	8	3	400	399	11	11	3	425	416
−2	8	3	270	257	13	11	3	327	305
0	8	3	477	479	−12	12	3	352	338
2	8	3	393	400	−10	12	3	365	363
4	8	3	442	436	−8	12	3	402	396
6	8	3	1097	1097	−6	12	3	223	241
8	8	3	637	626	−4	12	3	386	402
10	8	3	274	265	−2	12	3	287	290
12	8	3	217	223	0	12	3	429	415
14	8	3	454	451	4	12	3	277	264
16	8	3	784	767	−9	13	3	486	477
18	8	3	428	419	−7	13	3	754	791
−19	9	3	396	428	−5	13	3	1027	1041
−17	9	3	469	511	−3	13	3	460	474
−15	9	3	844	902	1	13	3	373	377
−13	9	3	578	562	3	13	3	1004	1018
−9	9	3	551	523	5	13	3	894	913
−7	9	3	1508	1471	7	13	3	560	579
−5	9	3	2289	2238	−26	0	4	1011	1014
−3	9	3	1079	1073	−24	0	4	501	487
−1	9	3	249	246	−22	0	4	199	208
1	9	3	996	1002	−20	0	4	362	359
3	9	3	2211	2186	−18	0	4	1578	1665
5	9	3	1847	1850	−16	0	4	837	831
7	9	3	983	966	−14	0	4	526	503
9	9	3	353	327	−12	0	4	244	191
11	9	3	641	646	−10	0	4	1393	1314
13	9	3	1146	1118	−8	0	4	2112	1974
15	9	3	721	723	−6	0	4	287	285
17	9	3	595	589	−4	0	4	1728	1677
−2	0	4	1710	1730	16	2	4	336	365
0	0	4	3242	3238	18	2	4	207	203

TABLE 1-continued

$MO_2CO_2S_4(CO)_2(NCCH_3)_2[S_2CH(C_2H_5)_2]_2$

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
2	0	4	2649	2604	22	2	4	271	273
4	0	4	1046	1052	24	2	4	233	244
6	0	4	311	297	−25	3	4	570	550
8	0	4	1879	1874	−23	3	4	240	223
10	0	4	2235	2295	−21	3	4	782	796
12	0	4	1325	1341	−19	3	4	1786	1827
14	0	4	1326	1345	−17	3	4	1763	1855
16	0	4	554	565	−15	3	4	684	660
18	0	4	1028	1038	−11	3	4	1314	1275
20	0	4	886	879	−9	3	4	2478	2459
22	0	4	482	491	−7	3	4	1847	1906
24	0	4	426	419	−5	3	4	1948	2045
−27	1	4	654	644	−3	3	4	2037	2003
−25	1	4	214	235	−1	3	4	400	405
−23	1	4	239	232	1	3	4	1837	1802
−21	1	4	571	570	3	3	4	2064	2121
−19	1	4	1249	1277	5	3	4	2581	2600
−17	1	4	1156	1214	7	3	4	2173	2211
−13	1	4	241	258	9	3	4	248	108
−11	1	4	823	779	11	3	4	388	387
−9	1	4	1926	1835	13	3	4	1358	1363
−7	1	4	1006	1024	15	3	4	1596	1596
−5	1	4	1002	994	17	3	4	567	580
−3	1	4	556	555	19	3	4	316	313
−1	1	4	903	884	23	3	4	422	420
1	1	4	849	814	−26	4	4	365	376
3	1	4	960	936	−22	4	4	351	353
5	1	4	3286	3229	−20	4	4	162	143
7	1	4	1171	1207	−18	4	4	409	422
9	1	4	171	176	−16	4	4	246	251
11	1	4	219	200	−14	4	4	389	357
13	1	4	807	816	−12	4	4	265	235
15	1	4	1096	1083	−10	4	4	159	121
17	1	4	447	442	−8	4	4	207	196
19	1	4	347	344	−6	4	4	1067	1113
23	1	4	241	236	−4	4	4	674	663
25	1	4	301	311	−2	4	4	204	206
−26	2	4	361	370	0	4	4	415	410
−24	2	4	241	229	2	4	4	317	318
−20	2	4	490	504	4	4	4	345	348
−18	2	4	665	693	6	4	4	124	154
−16	2	4	517	536	8	4	4	1695	1706
−10	2	4	1005	942	10	4	4	1052	1086
−8	2	4	608	582	12	4	4	185	151
−6	2	4	444	450	16	4	4	827	808
−4	2	4	370	363	18	4	4	1036	1004
−2	2	4	315	294	20	4	4	359	356
2	2	4	746	736	−25	5	4	539	527
4	2	4	924	933	−23	5	4	457	475
6	2	4	616	649	−19	5	4	495	504

TABLE I-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2									
H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
8	2	4	252	265	−17	5	4	902	944
10	2	4	671	659	−15	5	4	1028	1106
12	2	4	547	534	−13	5	4	467	501
14	2	4	388	383	−11	5	4	251	197
−9	5	4	1155	1175	−20	8	4	564	592
−7	5	4	1582	1654	−18	8	4	672	715
−5	5	4	1485	1518	−16	8	4	805	845
−3	5	4	275	262	−14	8	4	548	588
−1	5	4	756	769	−12	8	4	274	295
1	5	4	1040	1061	−10	8	4	639	631
3	5	4	2138	2161	−8	8	4	1300	1260
5	5	4	653	633	−6	8	4	1614	1577
7	5	4	216	199	−4	8	4	1078	1048
9	5	4	753	781	−2	8	4	134	59
11	5	4	1237	1196	0	8	4	291	290
13	5	4	718	710	2	8	4	1247	1232
15	5	4	401	383	4	8	4	1430	1423
19	5	4	349	333	6	8	4	946	944
21	5	4	392	363	8	8	4	905	899
23	5	4	300	309	10	8	4	159	60
−24	6	4	234	243	12	8	4	639	625
−20	6	4	545	570	14	8	4	815	804
−18	6	4	683	715	16	8	4	808	808
−16	6	4	429	472	18	8	4	778	754
−14	6	4	188	210	−19	9	4	637	698
−12	6	4	191	178	−17	9	4	899	958
−10	6	4	840	873	−15	9	4	728	782
−8	6	4	289	295	−13	9	4	235	242
−4	6	4	176	131	−11	9	4	564	555
0	6	4	586	589	−9	9	4	1310	1260
2	6	4	487	484	−7	9	4	1143	1113
4	6	4	454	446	−5	9	4	1011	992
6	6	4	478	467	−3	9	4	289	309
8	6	4	168	119	−1	9	4	537	516
10	6	4	706	701	1	9	4	1152	1148
12	6	4	569	552	3	9	4	1159	1170
14	6	4	594	574	5	9	4	940	935
16	6	4	392	376	9	9	4	458	446
18	6	4	302	315	11	9	4	773	777
22	6	4	367	344	13	9	4	527	535
−23	7	4	242	234	15	9	4	285	276
−21	7	4	350	371	−16	10	4	610	608
−19	7	4	863	907	−14	10	4	728	713
−17	7	4	648	701	−10	10	4	267	236
−13	7	4	306	312	−8	10	4	820	810
−11	7	4	531	466	−6	10	4	1391	1367
−9	7	4	1086	1044	−4	10	4	732	725
−7	7	4	628	641	−2	10	4	221	248
−5	7	4	355	348	0	10	4	188	174
−3	7	4	797	796	2	10	4	1043	1045

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−1	7	4	276	282	4	10	4	917	932
1	7	4	339	323	6	10	4	565	561
3	7	4	643	647	8	10	4	358	338
5	7	4	1011	1018	12	10	4	432	450
7	7	4	924	924	14	10	4	312	299
11	7	4	169	167	16	10	4	324	298
13	7	4	239	212	−15	11	4	419	426
15	7	4	539	518	−7	11	4	474	471
21	7	4	412	380	−5	11	4	319	321
−22	8	4	222	253	−3	11	4	254	243
−1	11	4	198	199	12	2	5	592	593
1	11	4	354	347	14	2	5	845	816
3	11	4	379	384	16	2	5	412	425
7	11	4	145	132	18	2	5	239	231
11	11	4	317	337	20	2	5	405	389
13	11	4	387	382	24	2	5	332	329
−12	12	4	285	280	−23	3	5	235	206
−10	12	4	411	424	−21	3	5	363	359
−8	12	4	377	403	−17	3	5	468	499
−6	12	4	336	343	−15	3	5	361	373
0	12	4	157	146	−13	3	5	228	197
2	12	4	342	356	−11	3	5	460	420
4	12	4	361	357	−9	3	5	250	241
6	12	4	255	282	−7	3	5	487	518
3	13	4	239	241	−5	3	5	663	660
−27	1	5	458	443	−3	3	5	299	309
−23	1	5	145	66	−1	3	5	1054	1068
−21	1	5	163	180	1	3	5	158	159
−19	1	5	839	843	3	3	5	579	586
−17	1	5	341	357	5	3	5	972	969
−13	1	5	178	163	7	3	5	1186	1206
−11	1	5	919	880	9	3	5	660	690
−9	1	5	1814	1734	13	3	5	560	562
−7	1	5	1547	1517	15	3	5	693	685
−5	1	5	527	554	17	3	5	520	498
−3	1	5	419	396	19	3	5	154	146
−1	1	5	3525	3398	21	3	5	296	283
1	1	5	3236	3142	−26	4	5	543	544
3	1	5	1492	1488	−22	4	5	375	379
5	1	5	296	300	−20	4	5	986	1015
7	1	5	2297	2323	−18	4	5	1429	1508
9	1	5	2544	2617	−16	4	5	937	989
11	1	5	1554	1575	−14	4	5	653	671
13	1	5	766	761	−12	4	5	413	421
15	1	5	332	313	−10	4	5	1771	1790
17	1	5	910	913	−8	4	5	2645	2713
19	1	5	700	691	−6	4	5	2404	2468
21	1	5	439	419	−4	4	5	1272	1300
23	1	5	446	458	−2	4	5	1264	1274
−26	2	5	245	239	0	4	5	2838	2911

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2									
H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−24	2	5	233	241	2	4	5	2500	2535
−22	2	5	221	201	4	4	5	1859	1911
−20	2	5	1045	1053	6	4	5	369	360
−18	2	5	1178	1191	8	4	5	1328	1349
−16	2	5	628	645	10	4	5	1213	1244
−14	2	5	885	860	12	4	5	1001	975
−12	2	5	323	314	14	4	5	603	591
−10	2	5	1435	1424	18	4	5	226	195
−8	2	5	2004	1967	20	4	5	254	242
−6	2	5	2069	2103	22	4	5	315	326
−4	2	5	2840	2720	−19	5	5	329	364
−2	2	5	1091	1031	−15	5	5	261	277
0	2	5	1759	1725	−11	5	5	854	892
2	2	5	1698	1729	−9	5	5	1115	1162
4	2	5	2610	2654	−3	5	5	753	771
6	2	5	1645	1695	−1	5	5	2333	2366
1	5	5	1869	1902	−6	8	5	1322	1299
3	5	5	611	595	−4	8	5	1082	1060
7	5	5	2012	2032	0	8	5	1018	1018
9	5	5	2045	2065	2	8	5	1002	979
11	5	5	1073	1074	4	8	5	979	977
13	5	5	466	440	6	8	5	233	232
15	5	5	351	354	8	8	5	435	432
17	5	5	1009	991	10	8	5	308	326
19	5	5	471	438	16	8	5	212	262
21	5	5	348	336	−19	9	5	594	609
−24	6	5	252	236	−17	9	5	897	946
−16	6	5	484	505	−15	9	5	844	854
−12	6	5	597	598	−13	9	5	348	293
−10	6	5	541	566	−11	9	5	803	775
−8	6	5	196	217	−9	9	5	1026	986
−4	6	5	1538	1591	−7	9	5	1554	1503
−2	6	5	908	927	−5	9	5	561	549
0	6	5	734	745	−3	9	5	224	170
2	6	5	587	589	1	9	5	778	781
4	6	5	341	372	3	9	5	1102	1076
6	6	5	936	947	5	9	5	709	696
8	6	5	635	626	7	9	5	659	643
10	6	5	981	969	9	9	5	306	297
12	6	5	667	654	11	9	5	607	606
16	6	5	168	169	13	9	5	535	513
18	6	5	437	439	15	9	5	488	476
20	6	5	640	621	17	9	5	407	401
22	6	5	298	296	−18	10	5	491	509
−23	7	5	256	268	−16	10	5	630	654
−21	7	5	491	524	−12	10	5	569	559
−19	7	5	689	713	−10	10	5	740	715
−17	7	5	489	499	−8	10	5	1078	1057
−15	7	5	260	243	−6	10	5	915	901
−13	7	5	284	288	−4	10	5	216	233

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−11	7	5	648	656	−2	10	5	898	901
−9	7	5	924	873	0	10	5	1174	1179
−7	7	5	1244	1174	2	10	5	1215	1219
−5	7	5	829	849	4	10	5	469	467
−3	7	5	297	324	6	10	5	347	334
−1	7	5	826	820	8	10	5	717	720
1	7	5	1240	1242	10	10	5	934	944
3	7	5	1159	1173	12	10	5	756	744
5	7	5	614	609	14	10	5	193	209
9	7	5	657	640	−11	11	5	243	212
11	7	5	740	704	−3	11	5	196	189
13	7	5	767	752	−1	11	5	330	333
15	7	5	453	446	1	11	5	228	243
17	7	5	195	164	3	11	5	142	194
19	7	5	201	214	7	11	5	303	304
−20	8	5	383	389	9	11	5	370	356
−18	8	5	755	808	11	11	5	176	186
−16	8	5	613	643	−12	12	5	373	364
−14	8	5	705	731	−8	12	5	163	148
−12	8	5	196	161	−6	12	5	200	186
−10	8	5	1089	1061	−4	12	5	401	399
−8	8	5	1104	1058	−2	12	5	224	201
0	12	5	187	171	−16	2	6	153	145
2	12	5	317	330	−12	2	6	900	871
10	12	5	419	404	−10	2	6	1028	1025
−7	13	5	1063	1074	−8	2	6	871	859
−5	13	5	518	518	−6	2	6	553	534
−1	13	5	436	458	−4	2	6	557	551
1	13	5	803	801	−2	2	6	1821	1856
3	13	5	770	783	0	2	6	1414	1459
−26	0	6	168	159	2	2	6	696	714
−22	0	6	259	264	4	2	6	205	201
−20	0	6	1055	1068	6	2	6	559	583
−18	0	6	651	649	8	2	6	729	767
−14	0	6	481	480	10	2	6	1059	1096
−12	0	6	1858	1781	12	2	6	672	669
−10	0	6	4346	4129	14	2	6	409	399
−8	0	6	1990	1879	16	2	6	285	265
−6	0	6	226	153	18	2	6	366	359
−4	0	6	1950	1962	20	2	6	532	525
−2	0	6	6397	6139	22	2	6	394	382
0	0	6	4640	4799	−23	3	6	149	150
2	0	6	2292	2342	−21	3	6	523	533
4	0	6	392	406	−19	3	6	1146	1181
6	0	6	3260	3334	−17	3	6	939	974
8	0	6	4099	4236	−15	3	6	1144	1207
10	0	6	2108	2128	−13	3	6	760	762
12	0	6	1057	1071	−11	3	6	1444	1455
16	0	6	1002	1001	−9	3	6	2482	2526
18	0	6	1154	1142	−7	3	6	1843	1928

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2									
H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
20	0	6	714	704	−5	3	6	2160	2210
22	0	6	890	877	−3	3	6	339	352
24	0	6	174	154	−1	3	6	1821	1868
−21	1	6	214	225	1	3	6	1703	1736
−19	1	6	545	531	3	3	6	1697	1729
−17	1	6	622	654	5	3	6	607	619
−15	1	6	711	696	7	3	6	794	301
−13	1	6	660	634	9	3	6	678	663
−11	1	6	932	903	11	3	6	301	279
−9	1	6	1305	1314	13	3	6	539	538
−7	1	6	1188	1178	17	3	6	248	244
−5	1	6	1548	1549	21	3	6	358	350
−3	1	6	250	235	23	3	6	321	331
−1	1	6	1990	2006	−22	4	6	285	201
3	1	6	879	904	−12	4	6	715	716
5	1	6	525	552	−10	4	6	1609	1648
7	1	6	178	203	−8	4	6	231	272
9	1	6	223	243	−4	4	6	958	994
11	1	6	175	154	−2	4	6	3027	3066
13	1	6	485	471	0	4	6	2054	2118
15	1	6	220	227	4	4	6	591	594
17	1	6	212	180	6	4	6	1629	1663
21	1	6	297	294	8	4	6	1830	1873
23	1	6	212	179	10	4	6	747	747
−24	2	6	164	124	16	4	6	669	652
−22	2	6	177	175	18	4	6	398	390
−20	2	6	287	293	22	4	6	316	305
−18	2	6	423	412	−23	5	6	245	261
−21	5	6	488	487	11	7	6	709	713
−19	5	6	604	644	13	7	6	224	205
−17	5	6	695	728	15	7	6	193	177
−15	5	6	198	184	19	7	6	400	383
−13	5	6	951	999	−20	8	6	396	426
−11	5	6	1261	1283	−18	8	6	553	588
−9	5	6	1355	1423	−16	8	6	683	685
−7	5	6	1132	1157	−14	8	6	307	292
−5	5	6	329	336	−12	8	6	353	364
−3	5	6	1642	1671	−10	8	6	563	545
−1	5	6	1763	1802	−8	8	6	1161	1114
1	5	6	1946	1959	−6	8	6	828	798
3	5	6	619	604	−4	8	6	278	272
5	5	6	585	567	0	8	6	686	683
7	5	6	1158	1171	2	8	6	1366	1346
9	5	6	1337	1338	4	8	6	682	679
11	5	6	1171	1158	6	8	6	464	440
13	5	6	302	301	10	8	6	716	727
15	5	6	269	271	12	8	6	791	791
17	5	6	478	489	14	8	6	317	319
19	5	6	639	631	16	8	6	171	156
21	5	6	406	399	18	8	6	330	322

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−24	6	6	192	140	−19	9	6	522	549
−20	6	6	446	475	−17	9	6	695	707
−18	6	6	326	336	−15	9	6	458	444
−14	6	6	176	128	−13	9	6	319	267
−12	6	6	967	971	−11	9	6	907	883
−10	6	6	1438	1419	−9	9	6	1309	1263
−8	6	6	1313	1355	−7	9	6	1238	1206
−6	6	6	609	622	−3	9	6	919	899
−4	6	6	808	805	−1	9	6	1391	1386
−2	6	6	2133	2161	1	9	6	1101	1119
0	6	6	2171	2178	3	9	6	508	498
2	6	6	1475	1503	5	9	6	373	376
4	6	6	481	478	7	9	6	860	848
6	6	6	1052	1051	9	9	6	764	760
8	6	6	1630	1612	11	9	6	478	478
10	6	6	1414	1395	13	9	6	168	157
12	6	6	1020	1005	15	9	6	266	247
14	6	6	538	526	−18	10	6	416	411
16	6	6	387	397	−16	10	6	220	251
18	6	6	561	548	−8	10	6	294	278
20	6	6	792	769	−6	10	6	151	146
−23	7	6	366	364	−4	10	6	184	177
−19	7	6	425	460	−2	10	6	473	472
−17	7	6	336	319	0	10	6	266	276
−15	7	6	932	963	6	10	6	356	337
−13	7	6	776	791	8	10	6	510	512
−11	7	6	371	370	−15	11	6	364	382
−9	7	6	557	536	−13	11	6	711	699
−7	7	6	683	649	−11	11	6	425	382
−5	7	6	1366	1318	−9	11	6	447	445
−3	7	6	300	286	−5	11	6	443	442
−1	7	6	206	207	−3	11	6	627	629
5	7	6	416	404	−1	11	6	648	661
9	7	6	501	474	1	11	6	933	936
3	11	6	544	552	14	2	7	459	483
5	11	6	235	221	16	2	7	539	519
7	11	6	376	385	18	2	7	480	471
9	11	6	897	897	20	2	7	689	679
11	11	6	960	957	22	2	7	410	393
−10	12	6	381	394	−23	3	7	214	221
−8	12	6	674	683	−17	3	7	197	198
−6	12	6	458	454	−15	3	7	160	44
−2	12	6	257	287	−13	3	7	281	279
0	12	6	475	473	−11	3	7	663	662
2	12	6	567	578	−9	3	7	518	514
4	12	6	243	256	−7	3	7	576	593
−3	13	6	206	213	−5	3	7	136	128
−1	13	6	214	150	−3	3	7	1036	1045
1	13	6	212	216	−1	3	7	134	99
−25	1	7	164	152	1	3	7	418	434

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−23	1	7	229	214	3	3	7	198	206
−21	1	7	773	778	5	3	7	300	292
−19	1	7	721	755	7	3	7	184	224
−15	1	7	287	299	9	3	7	257	259
−13	1	7	1836	1753	11	3	7	284	278
−11	1	7	2904	2833	15	3	7	160	42
−9	1	7	1092	1089	17	3	7	154	123
−7	1	7	302	253	19	3	7	314	315
−5	1	7	1076	1114	−22	4	7	406	409
−3	1	7	3429	3499	−20	4	7	781	796
−1	1	7	2714	2789	−18	4	7	557	560
1	1	7	1565	1642	−16	4	7	426	449
3	1	7	679	703	−14	4	7	552	593
5	1	7	724	730	−12	4	7	1307	1323
7	1	7	2078	2115	−10	4	7	1281	1289
9	1	7	1697	1723	−8	4	7	717	708
11	1	7	1464	1464	−4	4	7	878	894
13	1	7	1006	1024	−2	4	7	1426	1464
15	1	7	337	335	0	4	7	1322	1348
17	1	7	852	857	2	4	7	422	419
19	1	7	870	877	6	4	7	965	1010
21	1	7	905	893	8	4	7	1150	1183
23	1	7	321	316	10	4	7	756	743
−24	2	7	407	391	12	4	7	615	624
−20	2	7	614	611	14	4	7	248	281
−18	2	7	494	511	16	4	7	670	671
−16	2	7	729	751	18	4	7	726	722
−14	2	7	1208	1282	20	4	7	534	541
−12	2	7	690	685	−21	5	7	525	542
−10	2	7	771	768	−19	5	7	272	299
−8	2	7	407	415	−17	5	7	209	224
−6	2	7	1821	1824	−13	5	7	1159	1168
−4	2	7	307	326	−11	5	7	1845	1879
−2	2	7	341	336	−9	5	7	807	841
0	2	7	362	354	−5	5	7	1081	1098
2	2	7	461	444	−3	5	7	2337	2385
4	2	7	1229	1223	−1	5	7	2153	2189
6	2	7	214	207	1	5	7	651	672
10	2	7	155	94	3	5	7	664	664
12	2	7	870	868	5	5	7	459	450
7	5	7	1494	1491	16	8	7	211	187
9	5	7	1159	1141	−19	9	7	330	347
11	5	7	1176	1178	−15	9	7	220	260
13	5	7	1095	1087	−9	9	7	492	466
15	5	7	149	135	−7	9	7	263	257
17	5	7	569	562	−5	9	7	244	261
19	5	7	757	749	1	9	7	462	455
21	5	7	825	807	3	9	7	359	336
−22	6	7	443	451	5	9	7	385	381
−16	6	7	692	730	7	9	7	144	99

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−14	6	7	1044	1074	9	9	7	413	413
−12	6	7	555	530	13	9	7	475	461
−10	6	7	547	544	15	9	7	340	323
−8	6	7	619	587	−16	10	7	227	242
−6	6	7	1104	1122	−14	10	7	733	691
−4	6	7	1354	1396	−12	10	7	801	783
−2	6	7	1077	1073	−10	10	7	828	804
0	6	7	1294	1316	−8	10	7	500	492
2	6	7	403	383	−6	10	7	340	347
4	6	7	894	893	−4	10	7	961	957
6	6	7	820	811	−2	10	7	984	998
8	6	7	961	955	0	10	7	1288	1281
10	6	7	926	933	2	10	7	675	684
14	6	7	236	236	4	10	7	353	353
16	6	7	157	143	6	10	7	716	721
18	6	7	352	353	8	10	7	964	973
20	6	7	186	243	10	10	7	1066	1063
−21	7	7	239	280	12	10	7	393	402
−19	7	7	259	256	−9	11	7	314	319
−17	7	7	308	311	−7	11	7	211	243
−15	7	7	295	277	−3	11	7	252	249
−13	7	7	572	560	−1	11	7	258	271
−11	7	7	570	587	1	11	7	445	464
−9	7	7	798	752	3	11	7	450	464
−7	7	7	432	406	9	11	7	362	368
−3	7	7	353	354	−10	12	7	354	343
−1	7	7	1039	1002	−8	12	7	255	221
1	7	7	1311	1294	−6	12	7	269	240
3	7	7	739	724	−4	12	7	534	537
7	7	7	687	673	−2	12	7	575	586
9	7	7	1054	1041	0	12	7	946	936
11	7	7	1271	1271	2	12	7	490	529
13	7	7	454	468	6	12	7	286	290
17	7	7	586	587	−26	0	8	638	638
19	7	7	816	799	−24	0	8	668	642
−20	8	7	341	342	−22	0	8	1188	1184
−18	8	7	436	435	−20	0	8	582	578
−16	8	7	616	619	−18	0	8	788	809
−14	8	7	400	383	−16	0	8	1137	1175
−12	8	7	251	231	−14	0	8	2316	2373
−10	8	7	584	548	−12	0	8	2541	2449
−8	8	7	629	616	−10	0	8	157	88
−6	8	7	345	319	−8	0	8	522	502
−2	8	7	452	444	−6	0	8	1469	1416
0	8	7	185	164	−4	0	8	2818	2791
10	8	7	160	171	−2	0	8	1987	2059
0	0	8	861	849	−17	3	8	182	177
2	0	8	663	691	−15	3	8	182	160
4	0	8	240	245	−13	3	8	481	502
6	0	8	1909	1960	−11	3	8	237	243

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2									
H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
8	0	8	1465	1506	−9	3	8	904	925
10	0	8	1634	1603	−3	3	8	322	327
12	0	8	1202	1199	−1	3	8	578	590
14	0	8	311	301	1	3	8	301	295
16	0	8	1016	1041	3	3	8	717	760
18	0	8	657	650	5	3	8	463	458
20	0	8	776	771	7	3	8	811	830
22	0	8	240	243	9	3	8	597	594
−21	1	8	242	219	11	3	8	840	845
−19	1	8	316	321	13	3	8	191	168
−17	1	8	252	257	15	3	8	1095	1106
−15	1	8	414	438	17	3	8	1003	1004
−13	1	8	297	297	19	3	8	682	667
−11	1	8	542	561	21	3	8	279	308
−9	1	8	591	583	−24	4	8	219	215
−5	1	8	181	239	−22	4	8	625	629
−3	1	8	229	227	−20	4	8	296	273
−1	1	8	504	516	−18	4	8	529	540
3	1	8	307	310	−16	4	8	352	379
5	1	8	243	232	−14	4	8	942	951
7	1	8	586	579	−12	4	8	1293	1297
11	1	8	410	405	−8	4	8	199	209
13	1	8	158	161	−6	4	8	307	314
15	1	8	823	836	−4	4	8	1113	1130
17	1	8	506	526	−2	4	8	810	818
19	1	8	508	503	0	4	8	232	256
21	1	8	202	181	2	4	8	842	872
−26	2	8	223	203	4	4	8	377	398
−24	2	8	371	349	6	4	8	676	659
−22	2	8	354	359	8	4	8	367	335
−18	2	8	238	248	10	4	8	513	507
−16	2	8	412	421	12	4	8	993	1002
−14	2	8	529	544	16	4	8	179	179
−12	2	8	509	491	20	4	8	361	355
−10	2	8	442	437	−23	5	8	570	559
−8	2	8	159	133	−21	5	8	301	331
−6	2	8	263	224	−17	5	8	393	407
−4	2	8	344	352	−15	5	8	1073	1085
−2	2	8	1015	1010	−13	5	8	794	792
0	2	8	476	509	−11	5	8	1018	1021
2	2	8	893	909	−9	5	8	432	423
4	2	8	209	213	−7	5	8	921	953
6	2	8	601	625	−5	5	8	1199	1224
8	2	8	762	774	−3	5	8	1343	1359
10	2	8	801	798	−1	5	8	1167	1207
12	2	8	539	558	1	5	8	295	316
16	2	8	410	404	3	5	8	563	559
18	2	8	600	599	5	5	8	1218	1211
20	2	8	427	430	7	5	8	835	816
−23	3	8	164	190	9	5	8	920	909

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−21	3	8	396	411	11	5	8	164	158
−19	3	8	163	191	13	5	8	403	415
15	5	8	288	285	−11	9	8	187	201
17	5	8	542	543	−7	9	8	452	449
19	5	8	336	339	−5	9	8	499	490
−22	6	8	449	466	−3	9	8	462	467
−20	6	8	184	186	−1	9	8	299	299
−18	6	8	255	253	1	9	8	145	141
−16	6	8	637	619	3	9	8	352	329
−14	6	8	927	926	5	9	8	476	487
−12	6	8	868	871	7	9	8	418	406
−10	6	8	451	420	9	9	8	539	550
−4	6	8	859	834	13	9	8	165	160
−2	6	8	1324	1285	−6	10	8	520	508
0	6	8	833	802	−2	10	8	155	155
2	6	8	1351	1328	0	10	8	184	212
4	6	8	229	199	2	10	8	899	904
6	6	8	772	753	4	10	8	807	795
8	6	8	1250	1221	6	10	8	255	262
10	6	8	1509	1503	10	10	8	662	664
12	6	8	963	957	12	10	8	713	717
16	6	8	652	651	−13	11	8	581	554
18	6	8	865	867	−11	11	8	553	557
−19	7	8	188	207	−9	11	8	568	566
−15	7	8	476	473	−7	11	8	248	256
−13	7	8	444	472	−5	11	8	722	709
−11	7	8	550	554	−3	11	8	698	677
−9	7	8	977	957	−1	11	8	1117	1123
−7	7	8	174	177	1	11	8	673	678
−5	7	8	676	658	5	11	8	261	255
−3	7	8	687	680	7	11	8	753	758
−1	7	8	1389	1370	9	11	8	888	906
1	7	8	707	711	−8	12	8	202	201
-3	7	8	507	512	0	12	8	461	474
7	7	8	379	372	−25	1	9	710	688
9	7	8	532	549	−23	1	9	841	844
13	7	8	165	137	−21	1	9	199	241
15	7	8	346	348	−19	1	9	710	713
17	7	8	172	132	−17	1	9	878	892
−20	8	8	188	117	−15	1	9	1225	1219
−16	8	8	203	229	−13	1	9	1122	1118
−10	8	8	527	496	−11	1	9	235	232
−8	8	8	219	207	−9	1	9	323	339
−6	8	8	474	467	−7	1	9	242	219
−4	8	8	272	247	−5	1	9	1208	1231
−2	8	8	390	370	−3	1	9	1266	1278
0	8	8	789	798	−1	1	9	613	648
2	8	8	195	201	1	1	9	933	954
4	8	8	485	485	3	1	9	207	207
6	8	8	491	503	5	1	9	1154	1178

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
8	8	8	822	816	7	1	9	874	860
10	8	8	573	571	9	1	9	669	668
12	8	8	324	319	11	1	9	725	740
14	8	8	355	353	13	1	9	290	312
16	8	8	576	583	15	1	9	483	498
−17	9	8	313	304	19	1	9	288	327
−15	9	8	480	477	21	1	9	177	148
−13	9	8	479	479	−24	2	9	170	118
−20	2	9	775	767	−17	5	9	691	702
−18	2	9	705	701	−15	5	9	678	699
−12	2	9	978	978	−13	5	9	801	828
−10	2	9	1311	1299	−11	5	9	223	176
−8	2	9	226	185	−7	5	9	467	467
−6	2	9	284	305	−5	5	9	454	447
−4	2	9	166	133	−3	5	9	853	826
−2	2	9	213	223	−1	5	9	551	537
0	2	9	349	321	1	5	9	1425	1391
2	2	9	442	441	3	5	9	456	441
4	2	9	1032	1056	5	5	9	564	555
6	2	9	1509	1472	7	5	9	575	579
8	2	9	434	434	9	5	9	667	670
10	2	9	532	538	11	5	9	792	778
12	2	9	316	302	15	5	9	227	259
14	2	9	1475	1495	−22	6	9	184	198
16	2	9	830	832	−20	6	9	354	350
18	2	9	567	588	−16	6	9	746	764
20	2	9	298	281	−14	6	9	751	772
−19	3	9	180	167	−12	6	9	698	705
−17	3	9	204	207	−10	6	9	607	588
−9	3	9	221	174	−8	6	9	524	504
−7	3	9	251	240	−6	6	9	835	798
−5	3	9	155	158	−4	6	9	631	604
−1	3	9	185	185	−2	6	9	934	912
1	3	9	208	204	0	6	9	355	359
3	3	9	898	930	2	6	9	710	684
7	3	9	556	538	8	6	9	436	432
9	3	9	312	316	10	6	9	220	229
11	3	9	541	534	14	6	9	334	338
15	3	9	378	384	−19	7	9	297	285
17	3	9	465	485	−17	7	9	492	507
−24	4	9	452	444	−15	7	9	273	292
−22	4	9	199	183	−13	7	9	184	189
−20	4	9	441	436	−11	7	9	193	182
−18	4	9	883	889	−7	7	9	271	286
−16	4	9	822	843	−5	7	9	523	513
−14	4	9	458	460	−3	7	9	367	364
−12	4	9	228	200	−1	7	9	629	608
−10	4	9	921	913	1	7	9	222	226
−8	4	9	938	928	3	7	9	259	276
−6	4	9	1003	1037	5	7	9	429	451

TABLE 1-continued

$Mo_2Co_2S_4(CO)_2(NCCH_3)_2[S_2CH(C_2H_5)_2]_2$

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−4	4	9	1124	1155	7	7	9	904	906
−2	4	9	238	258	9	7	9	801	798
0	4	9	150	140	13	7	9	255	210
2	4	9	828	830	15	7	9	617	620
4	4	9	1261	1246	−18	8	9	488	500
6	4	9	993	979	−12	8	9	436	423
8	4	9	625	618	−10	8	9	918	899
12	4	9	595	601	−8	8	9	370	363
14	4	9	821	825	−2	8	9	503	487
16	4	9	790	802	0	8	9	243	235
18	4	9	331	318	2	8	9	271	276
−23	5	9	598	597	4	8	9	471	482
−21	5	9	188	178	6	8	9	293	285
−19	5	9	530	563	10	8	9	154	127
12	8	9	293	280	−9	1	10	634	622
14	8	9	439	433	−7	1	10	342	347
−17	9	9	311	292	−5	1	10	931	937
−15	9	9	426	432	−1	1	10	609	632
−9	9	9	497	490	1	1	10	282	292
−7	9	9	1112	1083	3	1	10	1326	1360
−5	9	9	498	491	5	1	10	1158	1159
−3	9	9	273	276	11	1	10	551	567
1	9	9	1143	1136	13	1	10	983	1004
3	9	9	1128	1142	15	1	10	356	356
5	9	9	578	571	17	1	10	242	263
7	9	9	427	414	−20	2	10	228	254
9	9	9	223	215	−18	2	10	386	381
11	9	9	701	698	−16	2	10	385	369
13	9	9	582	594	−14	2	10	485	445
−14	10	9	598	599	−8	2	10	297	259
−12	10	9	395	369	−6	2	10	574	588
−10	10	9	169	168	−4	2	10	370	403
−8	10	9	648	631	−2	2	10	249	235
−6	10	9	918	884	2	2	10	418	415
−4	10	9	559	558	4	2	10	381	359
−2	10	9	658	665	6	2	10	586	585
2	10	9	455	470	12	2	10	284	314
4	10	9	414	402	14	2	10	336	350
6	10	9	504	504	−21	3	10	780	777
8	10	9	513	516	−19	3	10	1052	1040
−7	11	9	265	274	−17	3	10	488	499
−5	11	9	263	286	−15	3	10	308	328
−1	11	9	374	367	−11	3	10	1063	1103
1	11	9	403	402	−9	3	10	966	956
3	11	9	383	368	−7	3	10	1271	1274
−24	0	10	689	670	−5	[illegible]	10	[illegible]447	1378
−20	0	10	771	779	−3	3	10	212	242
−18	0	10	870	848	−1	3	10	652	683
−16	0	10	831	818	1	3	10	1254	1224
−14	0	10	909	900	3	3	10	2069	2038

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
−12	0	10	202	212	5	3	10	1702	1689
−10	0	10	599	575	9	3	10	185	222
−8	0	10	416	419	11	3	10	799	809
−6	0	10	1285	1259	13	3	10	1207	1219
−4	0	10	1070	1101	15	3	10	641	662
−2	0	10	221	234	17	3	10	232	238
0	0	10	389	383	−22	4	10	187	178
2	0	10	475	483	−20	4	10	223	218
4	0	10	1544	1490	−18	4	10	201	186
6	0	10	520	529	−16	4	10	236	225
8	0	10	678	686	−14	4	10	448	467
10	0	10	225	214	−8	4	10	454	466
12	0	10	551	560	−4	4	10	454	459
16	0	10	509	500	−2	4	10	348	328
18	0	10	199	217	0	4	10	394	387
−21	1	10	464	458	2	4	10	428	422
−19	1	10	557	554	4	4	10	229	232
−17	1	10	199	226	6	4	10	677	659
−13	1	10	339	341	8	4	10	479	488
−11	1	10	713	705	14	4	10	401	401
16	4	10	720	722	0	8	10	797	792
18	4	10	242	241	2	8	10	926	937
−19	5	10	521	538	4	8	10	813	820
−17	5	10	859	877	6	8	10	720	729
−15	5	10	830	840	8	8	10	330	346
−13	5	10	301	338	10	8	10	428	432
−11	5	10	392	357	12	8	10	583	576
−9	5	10	786	784	−15	9	10	437	458
−7	5	10	1346	1299	−11	9	10	398	401
−5	5	10	661	632	−9	9	10	824	801
−3	5	10	329	291	−7	9	10	879	848
−1	5	10	315	295	−5	9	10	554	528
1	5	10	1219	1181	−1	9	10	492	487
3	5	10	586	555	1	9	10	753	749
5	5	10	386	395	3	9	10	674	681
7	5	10	243	230	5	9	10	357	360
9	5	10	496	484	9	9	10	333	346
11	5	10	402	391	11	9	10	371	349
13	5	10	221	237	−10	10	10	425	391
15	5	10	238	216	−8	10	10	799	770
−20	6	10	406	442	−6	10	10	373	356
−18	6	10	426	465	−2	10	10	232	232
−16	6	10	372	355	0	10	10	516	523
−14	6	10	539	561	2	10	10	544	567
−8	6	10	277	233	4	10	10	253	266
−6	6	10	796	763	6	10	10	261	224
−4	6	10	572	546	8	10	10	170	58
−2	6	10	327	345	−7	11	10	418	381
2	6	10	519	509	−5	11	10	351	338
4	6	10	565	578	−3	11	10	483	481

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
6	6	10	494	524	−1	11	10	273	238
12	6	10	331	324	1	11	10	161	164
14	6	10	336	365	3	11	10	210	128
19	7	10	405	391	−23	1	11	257	269
17	7	10	170	126	−21	1	11	216	223
15	7	10	191	211	−19	1	11	394	382
13	7	10	407	408	−17	1	11	569	570
11	7	10	855	860	−15	1	11	827	813
−5	7	10	293	277	−13	1	11	339	328
−3	7	10	352	345	−11	1	11	535	521
−1	7	10	519	499	−9	1	11	722	696
1	7	10	425	422	−7	1	11	1110	1121
3	7	10	694	679	−5	1	11	832	834
5	7	10	452	452	−3	1	11	697	693
7	7	10	301	287	−1	1	11	1031	981
9	7	10	290	249	1	1	11	1063	1029
11	7	10	257	249	3	1	11	924	921
13	7	10	423	418	5	1	11	538	521
18	8	10	502	497	7	1	11	938	940
16	8	10	542	560	9	1	11	660	678
14	8	10	179	144	11	1	11	553	565
10	8	10	542	529	15	1	11	328	343
−8	8	10	1016	985	17	1	11	287	302
−6	8	10	766	748	−22	2	11	281	267
−4	8	10	474	453	−20	2	11	606	590
−2	8	10	256	249	−18	2	11	419	401
−16	2	11	649	636	−5	5	11	555	514
−14	2	11	688	686	−3	5	11	550	529
−12	2	11	406	387	−1	5	11	626	614
−10	2	11	1037	1023	1	5	11	438	420
−8	2	11	1539	1534	3	5	11	436	426
−6	2	11	2132	2120	5	5	11	443	443
−4	2	11	805	777	7	5	11	889	897
−2	2	11	837	776	9	5	11	309	338
0	2	11	1058	1011	11	5	11	301	303
2	2	11	2332	2302	15	5	11	500	506
4	2	11	1530	1514	−18	6	11	302	290
6	2	11	336	337	−16	6	11	179	166
10	2	11	662	664	−10	6	11	191	197
12	2	11	1014	1036	−6	6	11	479	477
14	2	11	663	681	−4	6	11	277	261
16	2	11	263	293	2	6	11	359	359
18	2	11	229	247	4	6	11	463	464
−23	3	11	187	198	8	6	11	230	203
−17	3	11	214	186	10	6	11	183	159
−13	3	11	231	221	12	6	11	167	146
−9	3	11	673	672	−19	7	11	303	338
−7	3	11	233	215	−17	7	11	490	488
−3	3	11	363	367	−15	7	11	363	347
−1	3	11	333	291	−11	7	11	259	304

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
1	3	11	288	287	−9	7	11	684	681
3	3	11	405	403	−7	7	11	817	799
5	3	11	803	811	−5	7	11	642	625
7	3	11	359	386	−3	7	11	310	236
11	3	11	310	342	−1	7	11	625	629
13	3	11	519	539	1	7	11	881	876
15	3	11	442	445	3	7	11	922	921
17	3	11	270	239	5	7	11	568	549
−22	4	11	326	321	9	7	11	434	444
−20	4	11	681	672	11	7	11	587	604
−18	4	11	813	807	13	7	11	532	549
−16	4	11	878	873	−16	8	11	489	506
−14	4	11	279	261	−14	8	11	312	304
−12	4	11	542	560	−12	8	11	308	286
−10	4	11	1253	1255	−10	8	11	776	725
−8	4	11	1611	1584	−8	8	11	1026	1003
−6	4	11	1377	1352	−6	8	11	930	915
−4	4	11	478	453	−4	8	11	186	134
−2	4	11	943	934	−2	8	11	639	638
0	4	11	1665	1638	0	8	11	851	826
2	4	11	1490	1469	2	8	11	791	785
4	4	11	1172	1148	4	8	11	531	544
6	4	11	250	232	6	8	11	189	184
8	4	11	548	560	8	8	11	305	302
10	4	11	711	721	10	8	11	172	152
12	4	11	750	756	−13	9	11	320	294
14	4	11	357	321	−11	9	11	610	599
−17	5	11	176	154	−9	9	11	926	923
−15	5	11	695	718	−7	9	11	550	556
−13	5	11	378	350	−1	9	11	429	435
−9	5	11	355	359	1	9	11	759	774
−7	5	11	671	658	3	9	11	498	515
5	9	11	545	547	10	2	12	500	507
7	9	11	398	383	12	2	12	422	426
9	9	11	346	363	−21	3	12	534	509
10	10	11	579	559	−19	3	12	721	700
−8	10	11	680	666	−17	3	12	907	886
−6	10	11	374	392	−15	3	12	776	765
−2	10	11	341	344	−13	3	12	459	400
0	10	11	629	645	−11	3	12	1201	1226
2	10	11	556	561	−9	3	12	1412	1406
20	0	12	383	388	−7	3	12	1818	1771
18	0	12	443	397	−5	3	12	614	589
16	0	12	603	557	−3	3	12	702	690
12	0	12	1219	1196	−1	3	12	1095	1062
10	0	12	826	814	1	3	12	1483	1464
−8	0	12	841	846	3	3	12	1258	1267
−4	0	12	1490	1496	5	3	12	313	298
−2	0	12	1574	1561	9	3	12	448	462
0	0	12	1136	1090	11	3	12	859	871

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
2	0	12	405	405	13	3	12	611	634
4	0	12	717	700	15	3	12	192	202
6	0	12	1228	1243	−18	4	12	199	120
8	0	12	1042	1060	−16	4	12	312	298
10	0	12	597	623	−14	4	12	218	153
12	0	12	445	443	−12	4	12	343	358
14	0	12	321	320	−6	4	12	190	197
16	0	12	434	471	−4	4	12	955	936
21	1	12	354	312	−2	4	12	671	644
19	1	12	259	242	2	4	12	268	258
17	1	12	438	432	4	4	12	773	794
15	1	12	593	571	6	4	12	845	871
11	1	12	689	674	8	4	12	225	234
−9	1	12	875	874	14	4	12	425	419
−7	1	12	1142	1148	−19	5	12	500	485
−5	1	12	424	386	−17	5	12	378	358
−3	1	12	323	311	−13	5	12	327	347
−1	1	12	634	610	−11	5	12	748	730
1	1	12	1012	1009	−9	5	12	788	786
3	1	12	678	655	−7	5	12	308	292
5	1	12	402	382	−5	5	12	260	223
7	1	12	179	128	−3	5	12	705	690
11	1	12	564	571	−1	5	12	813	805
13	1	12	456	483	1	5	12	636	646
15	1	12	205	136	3	5	12	183	171
−20	2	12	199	172	5	5	12	267	292
−18	2	12	211	208	7	5	12	475	472
−16	2	12	329	297	9	5	12	614	612
−12	2	12	289	289	11	5	12	324	352
−10	2	12	587	582	−18	6	12	281	302
−8	2	12	511	524	−16	6	12	400	434
−6	2	12	429	421	−12	6	12	454	446
−4	2	12	360	330	−10	6	12	631	634
−2	2	12	515	501	−8	6	12	630	644
0	2	12	667	653	−6	6	12	479	467
2	2	12	588	571	−4	6	12	522	510
6	2	12	261	255	−2	6	12	867	857
8	2	12	314	326	0	6	12	786	787
2	6	12	735	738	−8	2	13	1042	1036
6	6	12	540	533	−6	2	13	858	834
8	6	12	504	517	−2	2	13	207	210
10	6	12	775	786	0	2	13	646	629
12	6	12	519	541	2	2	13	1190	1188
−17	7	12	354	341	4	2	13	769	771
−15	7	12	481	486	6	2	13	177	155
−11	7	12	417	391	10	2	13	896	892
−9	7	12	504	489	12	2	13	706	729
−7	7	12	723	721	−19	3	13	179	158
−5	7	12	354	356	−11	3	13	206	168
−3	7	12	257	247	−9	3	13	368	364

TABLE I-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
1	7	12	248	276	−7	3	13	290	315
3	7	12	541	526	−5	3	13	371	339
7	7	12	272	284	−1	3	13	435	424
9	7	12	247	234	1	3	13	464	462
−12	8	12	367	356	3	3	13	220	190
−10	8	12	779	757	5	3	13	259	265
−8	8	12	839	850	7	3	13	208	208
−6	8	12	406	404	9	3	13	229	250
−4	8	12	202	212	−20	4	13	457	456
−2	8	12	333	354	−18	4	13	441	410
0	8	12	932	935	−14	4	13	459	452
2	8	12	808	823	−12	4	13	773	789
4	8	12	700	692	−10	4	13	715	711
6	8	12	281	274	−8	4	13	635	606
8	8	12	453	489	−4	4	13	431	419
−11	9	12	566	566	−2	4	13	615	591
−9	9	12	795	762	0	4	13	623	613
−7	9	12	596	570	2	4	13	631	642
−3	9	12	447	467	8	4	13	514	527
−1	9	12	631	646	10	4	13	608	633
1	9	12	611	604	12	4	13	421	418
3	9	12	248	245	−15	5	13	240	261
0	10	12	274	227	−13	5	13	662	664
−21	1	13	407	391	−11	5	13	278	268
−15	1	13	356	358	−5	5	13	702	711
−13	1	13	1050	1026	−3	5	13	747	732
−11	1	13	603	601	−1	5	13	355	356
−9	1	13	281	281	1	5	13	432	431
−7	1	13	273	207	5	5	13	434	446
−5	1	13	944	928	7	5	13	479	466
−3	1	13	1088	1066	9	5	13	631	645
−1	1	13	668	638	11	5	13	678	674
1	1	13	397	423	−16	6	13	473	483
5	1	13	692	678	−14	6	13	223	241
7	1	13	604	617	−8	6	13	313	316
9	1	13	791	809	−6	6	13	440	442
11	1	13	799	831	−4	6	13	346	345
15	1	13	207	224	−2	6	13	567	574
−20	2	13	464	451	0	6	13	320	305
−18	2	13	556	518	2	6	13	311	327
−16	2	13	727	699	4	6	13	364	369
−14	2	13	213	211	6	6	13	520	547
−12	2	13	597	581	8	6	13	435	459
−10	2	13	624	627	−13	7	13	258	266
−11	7	13	520	514	8	2	14	521	531
−9	7	13	652	637	10	2	14	486	475
−7	7	13	255	281	12	2	14	177	134
−3	7	13	383	359	−15	3	14	235	249
−1	7	13	675	685	−13	3	14	270	275
1	7	13	817	827	−9	3	14	259	222

TABLE 1-continued

MO2CO2S4(CO)2(NCCH3)2[S2CH(C2H5)2]2

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
3	7	13	530	525	−7	3	14	397	374
7	7	13	447	451	−3	3	14	165	102
9	7	13	823	822	1	3	14	767	771
−12	8	13	298	279	3	3	14	284	326
−10	8	13	258	245	7	3	14	212	223
−8	8	13	395	418	9	3	14	562	584
−6	8	13	280	273	11	3	14	425	439
2	8	13	354	354	−18	4	14	264	278
4	8	13	172	169	−16	4	14	409	389
6	8	13	262	235	−14	4	14	546	547
−7	9	13	188	152	−10	4	14	480	483
−5	9	13	183	157	−8	4	14	186	222
−3	9	13	219	173	−6	4	14	415	406
−1	9	13	587	569	−4	4	14	354	330
−20	0	14	282	272	2	4	14	190	195
18	0	14	473	470	4	4	14	198	197
16	0	14	764	749	8	4	14	174	209
4	0	14	1155	1124	10	4	14	428	453
2	0	14	357	325	−17	5	14	427	426
0	0	14	264	316	−15	5	14	461	453
−8	0	14	384	415	−13	5	14	384	398
−6	0	14	884	871	−11	5	14	322	348
−4	0	14	1054	1032	−7	5	14	413	423
−2	0	14	265	282	−5	5	14	522	536
0	0	14	541	525	−3	5	14	541	553
2	0	14	266	269	−1	5	14	406	410
4	0	14	604	618	3	5	14	310	336
6	0	14	577	576	5	5	14	329	360
8	0	14	843	882	7	5	14	496	529
10	0	14	852	869	9	5	14	225	270
12	0	14	229	225	−14	6	14	449	455
−15	1	14	189	143	−12	6	14	250	268
−7	1	14	446	441	−6	6	14	311	302
−3	1	14	188	213	−4	6	14	446	426
1	1	14	566	550	−2	6	14	417	440
3	1	14	327	327	0	6	14	682	673
9	1	14	414	432	2	6	14	388	379
11	1	14	196	193	6	6	14	499	532
−16	2	14	336	320	8	6	14	745	778
−14	2	14	325	299	−13	7	14	195	179
−12	2	14	231	232	−11	7	14	415	417
−4	2	14	343	341	−7	7	14	431	417
−2	2	14	391	375	−5	7	14	428	411
0	2	14	500	497	−3	7	14	627	600
2	2	14	251	267	−1	7	14	394	391
6	2	14	436	420	1	7	14	256	250
3	7	14	360	332	−7	7	15	202	227
5	7	14	376	364	−5	7	15	323	322
−4	8	14	237	264	−3	7	15	553	538
−2	8	14	566	565	−1	7	15	449	428

TABLE 1-continued

$MO_2CO_2S_4(CO)_2(NCCH_3)_2[S_2CH(C_2H_5)_2]_2$

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
0	8	14	416	411	−14	0	16	395	401
−17	1	15	593	569	−8	0	16	578	602
−15	1	15	773	752	−6	0	16	1010	1026
−13	1	15	267	266	−4	0	16	468	474
−7	1	15	547	557	2	0	16	768	793
−5	1	15	761	764	4	0	16	461	461
−3	1	15	449	469	−7	1	16	396	386
−1	1	15	473	479	−5	1	16	241	230
1	1	15	253	212	1	1	16	383	367
3	1	15	489	496	3	1	16	591	594
5	1	15	552	585	−14	2	16	260	263
7	1	15	480	513	−8	2	16	355	362
9	1	15	494	490	−6	2	16	400	411
−12	2	15	329	305	−4	2	16	321	336
−8	2	15	352	343	−2	2	16	194	164
0	2	15	417	405	2	2	16	259	291
4	2	15	496	501	4	2	16	320	348
6	2	15	270	272	−11	3	16	166	109
8	2	15	244	279	−9	3	16	227	244
−11	3	15	205	161	−7	3	16	632	666
1	3	15	251	219	−5	3	16	438	457
5	3	15	186	179	−1	3	16	181	182
7	3	15	171	149	1	3	16	696	702
−16	4	15	334	347	3	3	16	849	874
−14	4	15	307	320	5	3	16	274	251
−10	4	15	176	106	−10	4	16	213	158
−8	4	15	243	246	−6	4	16	459	449
−6	4	15	431	454	−2	4	16	179	158
−4	4	15	271	295	0	4	16	253	234
2	4	15	358	376	2	4	16	194	215
4	4	15	440	454	−9	5	16	561	590
6	4	15	326	346	−7	5	16	422	432
8	4	15	209	195	−5	5	16	295	318
−13	5	15	226	196	−3	5	16	234	199
−7	5	15	358	334	−1	5	16	299	307
−5	5	15	584	561	1	5	16	312	326
−3	5	15	305	273	−9	1	17	518	537
−1	5	15	516	520	−7	1	17	758	808
1	5	15	342	346	−5	1	17	205	213
3	5	15	300	295	1	1	17	576	600
5	5	15	449	475	−10	2	17	339	336
−12	6	15	438	422	−8	2	17	964	986
−8	6	15	515	518	−6	2	17	595	624
−6	6	15	375	386	−2	2	17	221	259
−4	6	15	345	375	0	2	17	857	877
−2	6	15	390	414	2	2	17	917	927
0	6	15	235	210	−5	3	17	199	182
2	6	15	178	153	−1	3	17	233	235

Significantly, the parent $Mo_2S_4(Et_2NCS_2)_2$ moiety appears as an essentially intact unit in the cluster, with the Mo-Mo bond length decreased very slightly (from 2.814 Å to 2.783 Å) and the Mo-S-Mo bridge angles and bond lengths little changed. However, the dihedral angle between the MoS_1S_1' and $Mo'S_1S_1'$ planes has opened up from 147.9° to 164.4°, and the initially terminal Mo=S bonds have elongated from 2.09 Å to 2.316 Å as their role changes to a bridging μ_3 mode, bound to two Co atoms as well as the original Mo. The binding of two acetonitrile molecules also raises the overall cluster electron count to 60 e^-, the predicted number for a stable M_4 cluster with six M-M bonds.

EXAMPLE 6

Production of $(Bu_2NCS_2)_2Mo_2(\mu^3\text{-}S)_4Cu_2Cl_2$

A sample of $(i\text{-}Bu_2NCS_2)_2Mo_2S_4$ (100 mg, .137 mmol) was dissolved in 15 ml warm CH_3CN, and solid CuCl (54 mg, 0.548 mmol) added. The mixture was stirred for 3 minutes, filtered, and the filtrate allowed to stand for 1 hour. The resulting fine red-brown crystalline solid was filtered and dried (yield: 113 mg). Anal. Calc. for $C_{18}H_{36}N_2S_8Mo_2Cu_2Cl_2$: C, 23.33; H, 3.92; N, 3.02; S, 27.67; Mo, 20.70; Cu, 13.71; Cl, 7.65. Found C, 24.38; H, 3.83; N, 3.11; S, 26.82; Mo, 20.51; Cu, 12.94; Cl, 7.55.

A single crystal x-ray diffraction study was carried out on the product. The structure is illustrated in Figure 2.

EXAMPLE 7

Hydrotreating Catalyst

An amount of the Example 5 material (0.80 gm) was decomposed on a model feed (5% dibenzothiophene/decalin) at 350°C and 3150 KPa H_2 in a modified batch autoclave and gave an extremely high desulfurization rate. A crude zero order rate constant derived from operating data obtained between 2 and 4 hours was determined to be 160 x 10^{16} molecules DBT/g. cat. precursor/sec. This material was therefore found to be an effective HDS catalyst.

It is to be understood that the above disclosure is by way of specific example and that numerous modifications and variations of the invention are available to those of ordinary skill in the art without departing from the true spirit and scope of the invention.

Claims

1. A composition of matter containing a heterometallic thiocubane cluster having the Formula

$$(M_2^1 M_2^2 S_4)L_2^1 L_2^2 L_2^3$$

wherein:
M^1 is Re, V, Mo or W,
M^2 is Co, Cr, Cu, Ni or Fe,
L^1 is a bidentate sulfur and/or nitrogen bearing ligand, and
L^2 is optional but, if present, is a monodentate S, N, P or O donor ligand,
L^3 is selected from CO, a monodentate anion ligand such as a halide (preferably Cl), mercaptide or alkoxide, or another O-, N-, P-or S-containing monodentate donor ligand, and wherein $(M_2^1 M_2^2 S_4)$ forms said heterometallic thiocubane cluster.

2. The composition of claim 1 wherein M^1 is Mo or W.

3. The composition of claim 1 or claim 2 wherein M^2 is Co or Cu.

4. The composition of any one of claims 1 to 3 wherein L^1 is selected from the group of xanthate, dithiophosphinate, dithiophosphate, o-aminobenzenethiolate, and dithiocarbamate.

5. The composition of claim 4 wherein L^1 is S_2CNR_2 and wherein R is independently H or a hydrocarbyl group having from 1 to 12 carbon atoms.

6. The composition of claim 5 wherein each R is C_2H_5.

7. The composition of any one of claims 1 to 6 wherein L^2 is acetonitrile.

8 The composition of any one of claims 1 to 6 wherein L^2 is omitted and L^3 is selected from CO or Cl.

9. A method of making a composition of matter containing a heterometallic thiocubane cluster having the formula $(M_2^1 M_2^2 S_4)L_2^1 L_2^2 L_2^3$ wherein $(M_2^1 M_2^2 S_4)$ forms the heterometallic thiocubane cluster and wherein M^1 is Re, V, Mo or W, M^2 is Co, Cr, Cu, Ni or Fe, L^1 is a bidentate sulfur- and/or nitrogen-bearing ligand, L^2 is optional but if present is a monodentate S, N, P or O donor ligand, L^3 is selected from CO, a monodentate anion ligand such as a halide (preferably Cl), mercaptide or

alkoxide, or another O-, N-, P- or S-containing monodentate donor ligand, the method comprising adding a substantially stoichiometric amount of a low valent complex based on Co, Cr, Cu, Ni or Fe to a solution or slurry of $M_2^1 S_4 L_2^1$ and adding L^3, and optionally L^2.

10. A method as in claim 9 in which M_2^1 is W and L^1 is $[S_2CNR_2]$ wherein R is an H or a C_1 to C_{12} hydrocarbyl group.

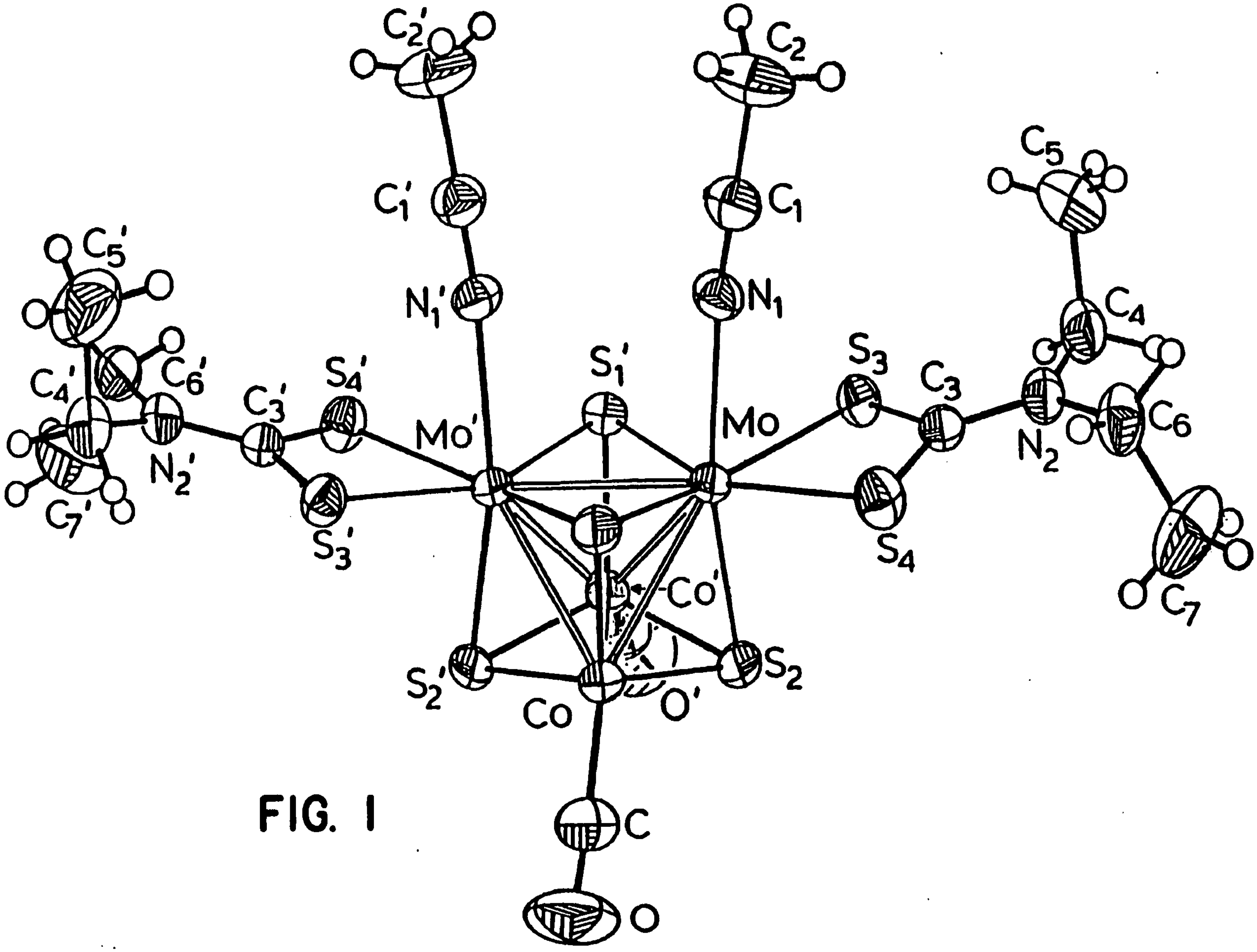
C2'
C2
C1'
C1
C5
C5'
N1'
N1
C4
S1'
S3
C4'
C6'
C3'
S4'
C3
Mo'
Mo
C6
N2'
N2
S3'
S4
C7'
Co'
C7
S2'
S2
Co
O'
C
O

FIG. 1

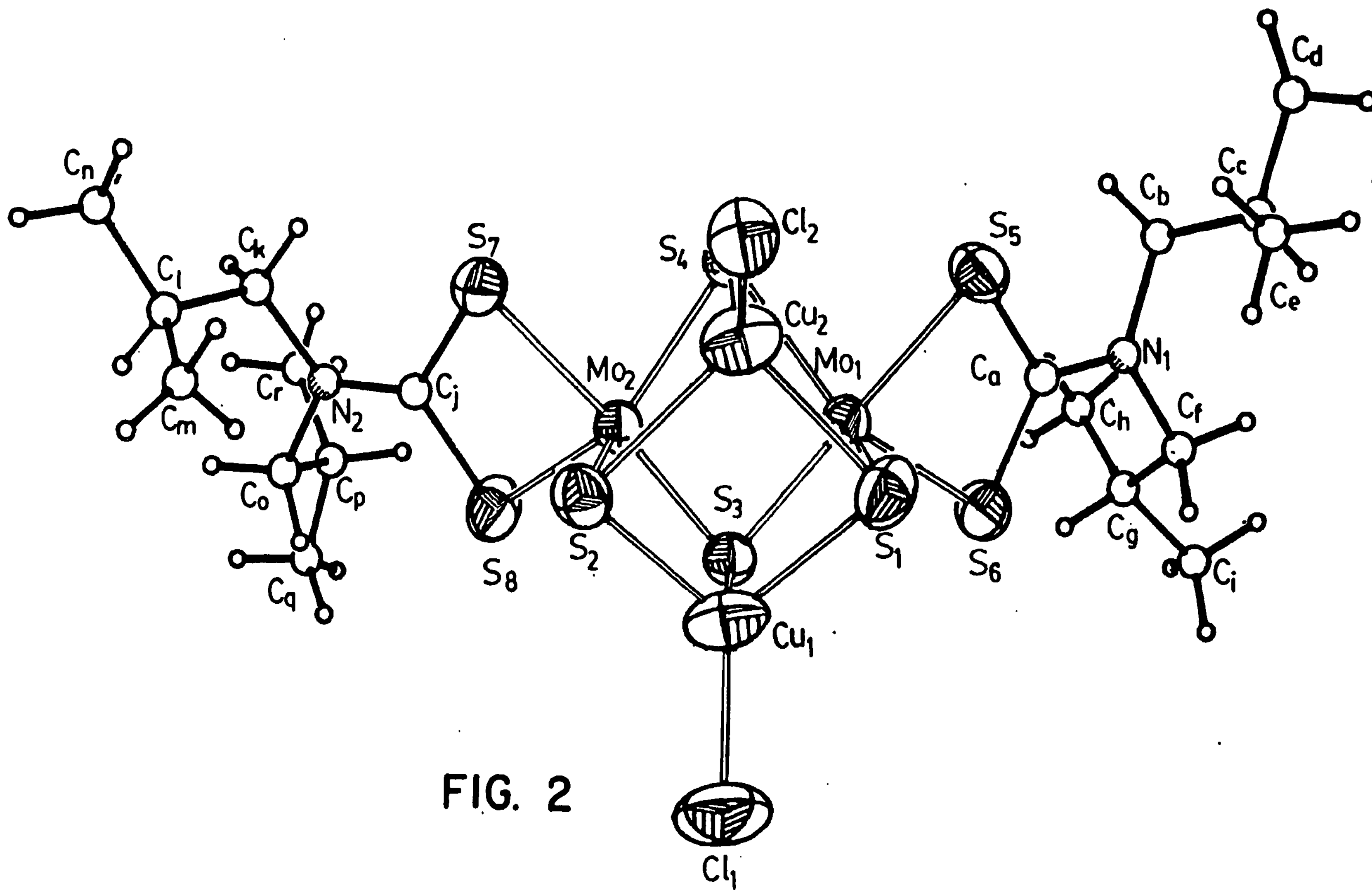
Cn
Cl
Ck
Cm
Cr
N2
Co
Cp
Cq
Cj
S7
S8
Mo2
S2
S4
Cl2
Cu2
Mo1
S3
S1
Cu1
Cl1
S5
S6
Ca
N1
Cb
Cc
Cd
Ce
Cf
Ch
Cg
Ci

FIG. 2

European Pa[illegible] Office

EUROPEAN SEARCH REPORT

Application Number

EP 87 31 0895

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
X	CHEMICAL ABSTRACTS, vol. 103, no. 12, 23rd September 1985, page 673, no. 97859t, Columbus, Ohio, US; T.R. HALBERT et al.: "Construction of heterometallic "thiocubanes" from M2S2(mu-S)2 core complexes: synthesis of Co2M2S4(S2CNEt2)2(CH3CN)2(CO)2 (M = Mo, W) and structure of the Co2Mo2(mu3-S)4 cluster" * Abstract * ---	1-10	C 07 F 11/00 C 07 F 15/06
A	INORG. CHEM., vol. 21, no. 4, 1982, pages 1699-1701, American Chemical Society; W.H. ARMSTRONG et al.: "Demonstration of the existence of single cubane-type MoFe3S4 clusters with S = 3/2 ground states: preparation, structure, and properties" * Page 1700, formula 2 * -----	1-10	

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

C 07 F 11/00
C 07 F 15/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	10-08-1988	PAUWELS G.R.A.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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L : document cited for other reasons

& : member of the same patent family, corresponding document

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